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COATINGS AND SURFACE TREATMENTS
FOR LONGTIME PROTECTION OF
Ti-8Al-1Mo-1V ALLOY SHEET FROM
HOT-SALT STRESS CORROSION

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SUMMARY

Results of an investigation to determine the effectiveness of several commercially feasible coatings and surface treatments in alleviating salt stress corrosion of Ti-8Al-1Mo-1V titanium-alloy sheet after longtime exposures at 600° F (590° K) in air at sea-level atmospheric pressure are presented. The coatings studied were electroplated nickel with a "black nickel" overcoat for high emittance, electrophoretically deposited aluminum, and a dipped coating of polyimide resin. The surface treatments included a vibratory treatment and glass-bead peening. The study included both residual-stress and self-stressed specimens which were dipped in a 3.4-percent NaCl solution, dried and exposed at 600° F (590° K) in circulating air ovens. Relative deflection measured in room-temperature bend tests or relative shortening measured in compression tests were used as quantitative indicators of stress-corrosion cracking in the specimens. An electron probe microanalyzer was used to determine interdiffusion between coating and substrate. The results indicated that the nickel and aluminum coatings were effective in preventing hot-salt stress corrosion for at least 10 000 hours exposure, whereas the polyimide coating peeled off the surface after 500 hours exposure. The glass-bead-peening process appeared to be effective in preventing or alleviating hot-salt stress corrosion for at least 10 000 hours. The vibratory treatment process proved to be effective in preventing or alleviating hot-salt stress corrosion for exposures of 15 000 hours when specimens were treated for at least 26 hours after fabrication.

INTRODUCTION

A significant problem with titanium alloys in supersonic transport applications is their susceptibility to salt stress corrosion at elevated temperatures. (See, for instance, refs. 1 and 2.) Salt-stress-corrosion cracking has been found in Ti-8Al-1Mo-1V alloy sheet at 450° F (500° K) after 6400 hours exposure and at 600° F (590° K) after 20 hours exposure for specimens with residual outer-fiber tensile stresses of 65 ksi (450 MN/m²)

(ref. 2). Load-induced tensile stresses of 50 ksi (350 MN/m^2) have been shown (ref. 1) to produce salt-stress-corrosion cracks in Ti-8Al-1Mo-1V alloy sheet after exposures at 550° F (560° K) for 500 hours.

A past study (ref. 3) has indicated that electroplated nickel and hot-dip or flame-sprayed aluminum coatings can provide stress-corrosion protection for titanium alloys for shorter times (up to 300 hours) but at higher temperatures (up to 1000° F (810° K)) than those required for supersonic transport skin materials. That same study indicated that steel shot peening and aluminum paints were ineffective in alleviating salt stress corrosion at 700° F (640° K) after 100 hours. The study was continued (ref. 4) and further research indicated that zinc coatings were also protective.

Another study (ref. 5) has investigated electroplated nickel and found it to be a promising coating for prevention of salt stress corrosion in a titanium alloy for temperature-time exposures of 850° F (730° K) and 2000 hours at a specimen stress (axial) level of 37 ksi (250 MN/m^2).

Several researchers (see, for instance, ref. 6) have investigated anodizing but have found that the anodic coating did not significantly affect the hot-salt stress corrosion of titanium alloys. However, an amorphous anodic coating has been shown to offer some protection. (See ref. 5.)

In another study (ref. 7) coatings of aluminum modified silicone, catalytically cured silicone, electrophoretically deposited aluminum, flame-sprayed aluminum, and zinc in a silicate vehicle were investigated. Preliminary results indicate that only the aluminum modified silicone provided good protection for the titanium alloy after exposures of 5000 hours at 500° F (530° K). The electrophoretic aluminum coating was not deposited on the edges of the test specimens in that investigation.

The duration of the test exposures in the investigations on coatings referred to in the preceding references were relatively short compared with the desired life associated with proposed commercial supersonic aircraft. The investigation described herein includes high-temperature exposures up to 15 000 hours which approach the expected cumulative exposures of 20 000 hours or more on future commercial supersonic aircraft. The effectiveness of several commercially available coatings and surface treatments in preventing or alleviating salt stress corrosion of titanium-alloy sheet at 600° F (590° K) was determined.

The units used for the physical quantities defined in this paper are given both in U.S. Customary Units and in the International System of Units (SI) (ref. 8). Factors relating the two systems are given in appendix A for the dimensions used herein.

STUDY OUTLINE

A flow chart outline of this study is shown in figure 1. Both residual-stress (surface tensile stress of 65 ksi (450 MN/m²)) and self-stressed (surface tensile stress of 50 ksi (350 MN/m²)) specimens of Ti-8Al-1Mo-1V alloy sheet were studied. Residual-stress specimens were fabricated by brake forming. Strips for self-stressed specimens were also brake formed, then two strips were spotwelded together at the ends to form one specimen as shown in figure 1. The coatings investigated included electroplated nickel with a "black nickel" overcoat for high-emittance, electrophoretically deposited aluminum, and a commercial polyimide-resin coating applied by dipping. The surface treatments investigated were vibratory cleaning and glass-bead peening. In general, coatings and surface treatments were applied to the specimens after fabrication; that is, after the specimen surfaces were under stress (an exception will be noted in appendix B for the vibratory cleaned specimens). Salt was applied by dipping the specimens into a 3.4-percent solution by weight of NaCl in water. The specimens were exposed in circulating laboratory air at 600° F (590° K). Relative deflection in a room-temperature bend test (residual-stress specimens) or relative shortening in a compressive test (self-stress specimens) was used as a quantitative indicator of stress-corrosion cracking in the specimens.

Metallurgical investigations included cross sectioning and electron probe micro-analysis of specimens. A special etch was used to confirm the existence of stress-corrosion cracks in some specimens. Preliminary results of this investigation were published in reference 9.

SPECIMENS, PROTECTIVE TREATMENTS, AND TEST PROCEDURES

Specimens

All specimens were fabricated from 0.050-inch (0.13 cm) thick Ti-8Al-1Mo-1V titanium-alloy sheet in the duplex annealed condition. Details of chemical composition, heat treatment, and typical tensile properties for the material used in this investigation are given in reference 6.

The configuration of the self-stressed specimen is shown in figure 2(a). Details of the fabrication and chemical cleaning processes and the analysis to determine the magnitude of the outer-fiber stresses are given in reference 6. All self-stressed specimens tested had an outer-fiber tensile stress of 50 ksi (350 MN/m²).

The configuration of the residual-stress specimen is shown in figure 2(b). Details of the fabrication process and the analysis to determine residual outer-fiber stresses are given in reference 2. All residual-stress specimens tested in this investigation were

brake formed over a 0.25-inch (0.6 cm) radius die, which produced a residual tensile stress of about 65 ksi (450 MN/m^2) on the outer fibers of the concave surface of the bend.

Protective Treatments

After fabrication and chemical cleaning, some specimens were coated with nickel, aluminum, or polyimide resin and others were subjected to surface treatments which included glass-bead peening or vibratory cleaning. Details of specimen preparation and coating or surface treatment application procedures are given in appendix B.

Salt Application

Salt was applied by dipping specimens into a 3.4-percent solution (by weight) of NaCl in distilled water at room temperature. This concentration of NaCl has previously been shown (ref. 2) to produce approximately the same magnitude of salt stress corrosion in Ti-8Al-1Mo-1V residually stressed specimens as a sea water dip. After dipping, the specimens were dried in an oven at 200° F (370° K) so that the salt would form a thin film of crystals on the surfaces subjected to tensile stresses. Photographs of salt-dipped self-stressed and residual-stress specimens are shown in figures 2(a) and 2(b), respectively.

The coatings, particularly the polyimide coating, provided poor wetting surfaces for the salt solution. In such cases, the salt coating was applied by placing droplets of the solution on the specimen surfaces with cotton swabs and allowing the droplets to dry. The thin salt coating was visually evident on the specimens when the water had evaporated.

Exposures

Salt-dipped and control (no salt) specimens were subjected to continuous heating in a circulating air laboratory oven at 600° F (590° K) $\pm 10^\circ \text{ F}$ (6° K). Specimens were removed from the ovens after selected exposure times up to 15 000 hours and mechanically tested.

Mechanical Tests

Compression tests.- After exposure, self-stressed specimens were subjected to room-temperature compression tests to determine quantitatively the effect which stress corrosion had on residual ductility. The equipment and procedures used in the testing and in generation of load-shortening curves for the specimens and the implication of the load-shortening curves in terms of stress-corrosion damage are described in reference 6.

Reverse-bend tests.- The residual-stress specimens were subjected to reverse-bend tests at room temperature after exposure. The equipment and procedures used in

these tests and in generation of load-deflection curves for these specimens are described in reference 2.

Metallurgical Examinations

Randomly selected residual-stress specimens were mounted on edge in bakelite after mechanical tests. The mounts were wet ground on a profile grinder to remove 0.12 inch (3.0 mm) from the edge of the specimen. The mounts were then successively wet ground on 240, 320, 400, and 600 grit silicon carbide papers and polished in a two-step vibratory polishing process. First polishing was accomplished with 5 micron ($5\mu\text{m}$) diamond particles on nylon cloth. Final polishing utilized 0.3 micron ($0.3\mu\text{m}$) levigated alumina on microcloth. A chemical etch in a solution of 97-percent water, 2-percent nitric acid, and 1-percent hydrofluoric acid, by volume, revealed microstructural constituents of the Ti-8Al-1Mo-1V alloy without severely attacking the thin nickel, aluminum, or polyimide coatings on the specimens.

Some of the specimens selected for metallurgical examination were subjected to analysis in an electron probe microanalyzer. The interfaces of nickel-coated and aluminum-coated specimens were analyzed by scanning the specimens at a rate of 5.3 micrometer/minute ($0.1\mu\text{m/s}$) under the electron beam using an accelerating voltage of 30 kV and a specimen current of $0.01\mu\text{A}$. X-ray signals from the elements Ti, Al, Ni, and V were autographically recorded as a function of distance across the specimen.

A chemical etch which can be used to indicate the existence of stress-corrosion cracks in titanium-alloy specimens has recently been utilized at the Langley Research Center. When using this etch, no preliminary preparation of the specimens, such as grinding or polishing, is necessary. The etch outlines the cracks and makes them easily visible at magnifications of $\times 50$ to $\times 100$. Specimens in this study which were suspected of having stress-corrosion cracks were subjected to this procedure, as follows. Uncoated specimens were immersed in a solution of 30 parts water, 60 parts hydrogen peroxide, and 10 parts hydrofluoric acid (by volume) for 2 minutes, rinsed in distilled water, dried and examined for cracks under a metallurgical microscope. If the specimens were nickel coated, this coating was mechanically removed (peeled off) from the surface of the specimen before the etch was applied.

RESULTS AND DISCUSSION

The magnitude of stress-corrosion effects or other embrittling effects on Ti-8Al-1Mo-1V alloy specimens are shown in the following sections as plots of relative deflection or relative shortening as a function of exposure time (plotted on a semilogarithmic scale) for each coating or surface treatment. For this study relative deflection

is defined as the ratio of deflection immediately prior to fracture for residually stressed specimens with coatings or surface treatments (most of which have been salt dipped and exposed) to the average deflection immediately prior to failure for uncoated, unexposed Ti-8Al-1Mo-1V alloy specimens. Relative shortening is similarly defined for self-stressed specimens. Lower values of relative deflection or relative shortening indicate more embrittlement of the specimens.

Nickel Coating

Relative deflection of residual-stress specimens with the nickel coating is shown in figure 3(a) for exposure times up to 10 000 hours at 600° F (590° K). The solid line in figure 3(a) (from ref. 2) indicates the behavior of salt-dipped specimens which have not been nickel coated. Considerable stress-corrosion effects for these exposure conditions are evidenced by a reduction of relative deflection to approximately 50 percent after 50 hours exposure and to 25 percent after 300 hours exposure. The dashed curve (also from ref. 2) indicates the behavior of exposed Ti-8Al-1Mo-1V alloy specimens which had not been nickel coated or salt dipped. Some reduction in relative deflection for these specimens after long exposures is evident. The circular data points in figure 3(a) indicate relative deflection of nickel coated control specimens which were not salt dipped. The square data points indicate relative deflection of nickel coated and salt-dipped residual-stress specimens. The close proximity of the circular and square data points and the dashed line indicated that no stress corrosion has occurred. The 10 000-hour data which indicate some embrittlement are discussed in a subsequent paragraph.

In figure 3(b) the relative shortening of nickel-coated self-stressed specimens is plotted. Symbols and curve designations are identical to those in figure 3(a) but, in figure 3(b), the solid and dashed curves represent unpublished data obtained at the Langley Research Center. Although more variability is evident in the data of figure 3(b) than in that of figure 3(a), the general trend again indicates that no stress corrosion has occurred. The 10 000-hour data are discussed subsequently.

Photographs of mechanically tested nickel coated residual-stress specimens are shown in figure 4 for both salt-dipped specimens and control specimens; figure 5 shows photographs of nickel-coated self-stressed specimens after testing. Of interest in these specimens was the failure zone. For exposures up to about 2000 hours, the nickel coating peeled off the titanium-alloy substrate at failure. After exposures longer than 2000 hours, the coating cracked at failure but was more adherent to the substrate. This result does not imply that the nickel coating did not adhere well to the substrate during the shorter exposures. On the contrary, adherence for all exposures was good and the coating exhibited considerable ductility in the mechanical tests before failure. At failure, however, the peeling associated with short exposure and the no peeling associated with

longer exposure were noted and may indicate the existence of some bonding changes in the substrate-coating interface with increased exposure. This result was investigated further, as noted in the following paragraphs.

Cross sections of edges of nickel-coated residual-stress specimens before and after exposure are shown in figure 6. No evidence of gross changes in microstructure or of the formation of visible intermediate phases at the coating-substrate interface was evident in the microscopic examination of these specimens, although the increased sharpness of the coating-substrate interface after the longer exposures may be further evidence of the phenomenon described in the previous paragraph.

Results of the electron probe analysis of the nickel-coated specimens are shown in figure 7. Relative percentages of 4 elements: nickel (which was only in the coating before exposure) and titanium, aluminum, and vanadium (which were only in the substrate before exposure) are plotted as a function of distance from the coating-substrate interface as visually determined during the electron probe scans. The as-coated curves in figure 7 have an S-curve shape which should not be misinterpreted. The interface was optically sharp; the apparent width of the interface is simply an indication that the characteristic X-rays emitted from the specimen emanated from a spot which was several micrometers in diameter. Scans after 2015 hours exposure produced curves (not plotted in fig. 7) which were almost identical to the as-coated curves; thus, no detectable diffusion had taken place during that exposure. After 7000 hours exposure at 600° F (590° K), however, the displacement of the exposure curves in figure 7 from the as-coated curves indicates that a detectable amount of diffusion has taken place.

This information correlates with the photographs of figures 4 and 5. Interdiffusion between substrate and coating could produce stronger bonding at the interface. The reduction of relative deflection (fig. 3(a)) and relative shortening (fig. 3(b)) for nickel-coated specimens after 10 000 hours exposure suggests limited usefulness of this coating after very long exposures. A close study of figure 3 indicates that the apparent reduction in ductility of the Ti-8Al-1Mo-1V alloy sheet was not caused by salt-stress-corrosion cracking, since both control specimens and salt-dipped specimens showed similar magnitudes of decrease in relative deflection and relative shortening. This result was confirmed by cross sectioning and by use of the special etchant which indicates the existence of stress-corrosion cracks. No such cracks were noted in these examinations.

If embrittlement is occurring in the nickel-coated specimens after 10 000 hours exposure, an explanation for embrittlement is suggested, based on the diffusion between coating and substrate described previously. After significant diffusion has taken place, the possibility of interface hardening due to formation of an extremely thin brittle phase or some other mechanism is likely after 10 000 hours exposure at 600° F (590° K). This hardened interface could then initiate cracks in the substrate beginning at the interface

when the material is deformed in the reverse-bend tests or compression tests, and could result in reduced ductility of the substrate.

High emittance in a coated titanium skin structure is an important requirement for supersonic transport applications. Low-emittance coatings will radiate less of the aerodynamic heating flux and will thus cause the skin to operate at a higher equilibrium temperature than a material with a high-emittance coating if other factors are equal. Emittance values for nickel coatings are given in appendix C. The nickel plating without the overcoat exhibited very low-emittance values, below 0.1. The "black nickel" overcoat was investigated as a possible means of raising these values. The emittance was raised to values well above 0.9 by the overcoat; these values prove the effectiveness of the black nickel which is so thin that the weight penalty due to its application is small.

In summary of the results for nickel-coated specimens, the nickel coating appears to provide good protection for Ti-8Al-1Mo-1V alloy sheet against salt stress corrosion at 600° F (590° K) for at least 10 000 hours exposure. However, a possible coating-substrate reaction which embrittles the sheet may be occurring after longer exposures due to solid-state diffusion. The low emittance of the nickel plating can be easily raised to emittance values approaching 1.0 by application of the black nickel overcoat. The procedures for nickel plating and the black nickel overcoat are standard, relatively inexpensive, commercial processes.

Aluminum Coating

Figures 8(a) and 8(b) show relative deflection and relative shortening values for aluminum-coated Ti-8Al-1Mo-1V alloy stress-corrosion specimens. Symbol and curve designations are similar to those of figures 3(a) and 3(b). The as-coated specimens showed a small reduction in ductility, but no evidence of stress corrosion was apparent after exposures of 10 000 hours at 600° F (590° K) for residual-stress specimens. However, as noted in appendix B, stress relaxation was detected in the self-stressed specimens as a result of the temperature-time exposures employed in the aluminum coating sintering process. Consequently, the stress level existing in the self-stressed specimens (fig. 3(b)) was approximately 20 percent of the original load-induced tensile stress. Although this sintering did not appear to affect substrate ductility, further study of the effect of sintering on the properties of the titanium-alloy substrate appears to be necessary if aluminum-coated Ti-8Al-1Mo-1V alloy sheet is to be considered for structural applications.

Photographs of tested specimens are shown in figures 9 and 10. The mottled appearance of the coating surface may be due to impurities in the suspension which were electrophoretically deposited along with the aluminum and which migrated to the specimen surface during the liquid-phase sintering process. This coating exhibited extremely good

adherence and ductility after all exposures investigated, as indicated by the lack of peeling in the coating after mechanical tests, shown in figures 9 and 10.

Cross sections of residual-stress specimens are shown in figure 11. The appearance of the specimens in these photomicrographs should not be misinterpreted. The darker areas in the coating near the interface and near the outer edge of the coating are due only to metallograph light shadowing effects of the substrate because of difficulties inherent in the polishing of a thin, soft coating on a harder substrate. No evidences of microstructural changes due to exposure were noted in substrate, coating, or interface in the microscopic examination of these specimens.

Figure 12 shows the results of electron probe analysis of the coating-substrate interfaces of aluminum-coated residual-stress specimens. The as-coated curves for Al, Ti, and V in figure 12 may indicate that some diffusion has occurred during the liquid-phase sintering process. Scans after 2000 hours exposure at 600° F (590° K) produced curves (not plotted in fig. 12) which were almost identical to the as-coated curves; thus, no detectable diffusion had taken place during that exposure. After 7000 hours exposure, the displacement of the exposure curves in figure 12 from the as-coated curves indicates that a detectable amount of diffusion has taken place. However, possibly since aluminum is already in the Ti-8Al-1Mo-1V alloy as an alpha stabilizer, the addition of more aluminum near the coating-substrate interface by diffusion apparently does not significantly affect the substrate ductility after exposures up to at least 10 000 hours (fig. 8).

In summary of the results for the aluminum-coated specimens, the aluminum coating appears to be effective in preventing stress corrosion in Ti-8Al-1Mo-1V alloy sheet for at least 10 000 hours at 600° F (590° K), based on results obtained for the residual-stress specimens. These results and the applicability of the electrophoretic deposition process for uniformly coating large, complex parts and the elimination of isostatic pressing from the coating procedure suggest the electrophoretic aluminum coating as a possible method for prevention of hot-salt stress corrosion in titanium alloys. However, the effects of the liquid-phase vacuum sintering process on the properties of the titanium-alloy substrate (discussed in appendix B) should be studied further.

Polyimide Coating

Exposures at 600° F (590° K) caused significant degradation of the polyimide-resin coating. Photographs of coated specimens are shown in figures 13 and 14. In the as-coated condition, the polyimide coating showed good adherence to the substrate and also exhibited remarkable ductility. Although the Ti-8Al-1Mo-1V alloy substrate had fractured under it, figures 13 and 14 show that before exposure the coating did not fail in the mechanical tests. The cracks in the substrate are visible through the brown transparent coating. With increasing exposure time up to 750 hours, however, the coating

became progressively more brittle as indicated by the appearance of exposed specimens in figures 13 and 14. Between 750 and 1000 hours the polyimide coating turned an opaque orange color and began to peel off the specimens in the exposure oven. Before 750 hours the salt had no apparent effect on the coating, but after the coating became opaque the areas containing salt appeared to peel off and degrade more quickly than the other areas of the specimen. (See fig. 13 for the 1000-hour exposures.)

The relative deflection of residual-stress specimens is presented in figure 15(a). No stress corrosion was noted for the polyimide-resin-coated specimens with the original salt dip, even when the coating had degraded. After exposure for 2000 hours, a group of specimens from which the coating had peeled were redipped in the salt solution. Further exposures of 300 to 1000 hours produced significant stress corrosion, and reduced relative deflection to approximately the same magnitude as that for uncoated specimens exposed for 80 to 300 hours. (See fig. 15(a).) Figure 15(b) shows relative shortening of polyimide-coated self-stressed specimens. In this case no specimens were redipped in the salt solution and even though the coating had peeled off parts of the specimen before 1000 hours exposure, no stress corrosion of the Ti-8Al-1Mo-1V alloy was noted after 1000 hours exposure.

It appears that the coating was effective in preventing stress corrosion up to at least 500 hours exposure. In peeling off the specimens after longer exposures, the polyimide apparently removed all salt from the specimens, and allowed no salt to remain on the titanium-alloy surfaces. Stress corrosion was noted in the specimens only after additional salt was applied.

Cross sections of polyimide-resin-coated residual-stress specimens are shown in figure 16. The problems of polishing and photographing a soft coating on a hard substrate were noted for the aluminum coating in the preceding section and also are applicable to the polyimide coating. No obvious changes in the coating are noted for exposures up to 500 hours (figs. 16(b) and 16(c)) but the cracks and irregularities in the coating after 1000 hours exposure (fig. 16(d)) indicate the degrading effects of this longer exposure.

The results discussed in the preceding paragraphs lead to a dual interpretation of polyimide degradation during exposure at 600° F (590° K) in air at sea-level atmospheric pressure: (1) continuing polymerization of the polyimide will tend to embrittle the polymer, cause the progressively more brittle failures in the mechanical tests after increasing exposure times, and ultimately cause the coating to peel off the specimens; (2) polyimides show significant oxidation effects during high-temperature exposures in the presence of oxygen (see, for instance, refs. 10 and 11) with volatile oxide formation. Oxidation proceeding at the coating-substrate interface after small defects have appeared in the coating would tend to decrease adherence and promote peeling. Reference 11 indicates that oxidation at 450° F (500° K) proceeds much more slowly than at 600° F

(590° K) and that oxidation at 600° F (590° K) proceeds more slowly at lower air pressures (35 torr (4.6 kN/m²)). These data may indicate that the polyimide coating would degrade much more slowly at the temperatures and pressures which a supersonic transport skin would encounter during cruising flight than it did at the higher temperatures and pressures of this investigation.

Emittance of polyimide-resin-coated Ti-8Al-1Mo-1V alloy sheet was measured as described in reference 12. The polyimide coating increased emittance of the titanium from approximately 0.17 to 0.7 (appendix C). This emittance increase could result in a measurable reduction of equilibrium temperatures of skin panels in high-temperature aircraft structures.

In summary of the results for polyimide-resin-coated specimens, the coating was adherent and ductile as applied by a simple dipping process and provided good protection against salt stress corrosion for at least 500 hours at 600° F (590° K). However, the coating became progressively more brittle with increasing exposure time and peeled off the specimens after 750 to 1000 hours exposure. Thus, the polyimide coating shows limited applicability at 600° F (590° K) and sea-level atmospheric pressure but may be considered for lower temperature and lower pressure exposures, where its protection lifetimes should be considerably longer.

Glass-Bead Peening

In presenting preliminary results of this investigation, references 9 and 13 suggest that the effectiveness of the glass-bead-peening treatment in alleviating stress corrosion of the Ti-8Al-1Mo-1V alloy sheet may be diminishing after approximately 3000 hours exposure at 600° F (590° K). Relative deflection and relative shortening data, presented in figures 17(a) and 17(b), for longer exposure times indicate that the peening treatment is effective. After 3000 hours exposure, relative deflection and relative shortening were down 30 to 50 percent from as-fabricated (before exposure) values but after exposures up to 10 000 hours relative deflection and relative shortening values increased to within 70 to 80 percent of that of unexposed specimens with no peening.

Several 3000-hour-exposure specimens were examined after the special etchant (to reveal salt-stress-corrosion cracks) had been applied but no evidence of stress-corrosion cracking was noted. The lowering of relative deflection and relative shortening for exposures to 3000 hours, followed by an apparent increase in ductility of the specimens for increasing exposure times to at least 10 000 hours, is not explained by the results of this investigation.

Photographs of tested glass-bead-peened specimens are shown in figure 18 for residual-stress salt-dipped and control specimens and in figure 19 for self-stressed specimens. No significant changes in specimen surfaces were noted as a result of the

exposures. Photomicrographs of cross sections of glass-bead-peened residual-stress specimens are shown in figure 20. No apparent changes in the microstructure were caused by the peening process or by exposures up to 10 000 hours at 600⁰ F (590⁰ K).

The possibility of stress relaxation after long elevated-temperature exposures which might tend to relieve the compressive stresses induced by glass-bead peening has often been suggested as a limiting factor in the use of peening to alleviate stress corrosion. A recent study, reference 14, using X-ray diffraction methods to measure the residual compressive surface stresses in titanium-alloy sheet which resulted from glass-bead peening, indicates that the peening process used in the present investigation applied compressive stresses of approximately 110 ksi (760 MN/m²) at the surface and to a depth of 0.0007 inch (18 μm) below the surface of the titanium alloy. That study also showed that the effect of 1000 hours of exposure at 600⁰ F (590⁰ K) could reduce these residual stresses by 60 percent. If a first approximation is made that the peening-induced compressive stresses are algebraically additive with the fabrication-induced tensile stresses on the surfaces of the stress-corrosion specimens of this investigation, a residual compressive stress on the order of 45 to 60 ksi (350 to 410 MN/m²) would be present on the residual-stress and self-stressed specimen surfaces, respectively, after the glass-bead-peening process. During exposure, the magnitude of the compressive stresses should decrease but even after exposures from 1000 to 10 000 hours it would be expected that the stresses would remain sufficiently compressive on all outer fibers of the specimen to prevent stress corrosion as was apparently indicated by the results shown in figures 17(a) and 17(b) after 10 000 hours exposure.

In summary of the results for the glass-bead-peened specimens, this procedure appears to be effective in preventing or alleviating salt stress corrosion after exposures up to at least 10 000 hours at 600⁰ F (590⁰ K) although some loss of ductility in the material after 3000 hours exposure and the subsequent increases in ductility after further exposure is not definitively explained. The glass-bead-peening process is simple and relatively inexpensive and is applicable for both small complex shapes and large areas of a structure. These factors suggest that glass-bead peening may be seriously considered for parts subjected to environments in which hot-salt stress corrosion is expected.

Vibratory Cleaning

The previously described data for each of the coatings and surface treatments showed that residual-stress and self-stressed specimens produced similar data in the mechanical tests; that is, relative deflection values for a given coating and exposure time were similar to relative shortening values. This similarity of data was not found with vibratory-cleaned specimens as shown in figure 21(a) and 21(b). Figure 21(a) shows relative deflection values for Ti-8Al-1Mo-1V alloy residual-stress specimens. The

possibility of stress-corrosion effects is noted after 6000 hours exposure and definite indications of stress corrosion are shown by the difference in relative deflection values between the control specimen and the salt-dipped specimens after 8000 hours exposure. Surfaces of residual-stress salt-dipped specimens which had been exposed for various times were subjected to the special etchant which reveals cracks. These specimens were not mechanically tested. No cracking was noted for the as-fabricated and 4000-hour-exposure specimens. Cracks were readily visible under stereoscopic microscopic examination at 75 diameters magnification in specimens after 6000 and 8000 hours exposure. After a reverse bend test, the cracks have opened, as shown in figure 22.

Relative shortening values for vibratory-cleaned self-stressed specimens are shown in figure 21(b). No evidence of salt-stress-corrosion effects were noted for these specimens after exposures of 15 000 hours at 600° F (590° K), which is contrary to the results obtained for the residual-stress specimens noted in the preceding paragraph.

Photographs of tested residual-stress salt-dipped and control specimens are shown in figure 23; photographs of tested self-stressed specimens are shown in figure 24. No significant changes in specimen surfaces were noted after exposure. Photomicrographs of cross sections of tested residual-stress specimens are shown in figure 25. Figure 25(a) is typical of all vibratory-treated residual-stress specimens exposed in this study. Figure 25(b) is a residual-stress specimen which has been vibrated 26 hours after fabrication so that the cross section is typical of the treatment given the self-stressed specimens of this study before exposure. Exposed residual-stress specimens are shown in figures 25(c) and 25(d). No significant microstructural effects were noted in any vibratory-cleaned specimen cross sections.

The primary reason for the appearance of stress corrosion in the vibratory-cleaned residual-stress specimens appears to be the relatively short time (4 hours) that these specimens were subjected to the vibratory treatment. Reference 14 indicates that residual compressive stresses due to the length of treatment are approximately 40 ksi (280 MN/m²). The residual tensile stress on the outer fiber of the concave surface of the specimen is approximately 65 ksi (450 MN/m²). If the first approximation of algebraically additive stresses is made, a tensile stress of 25 ksi (170 MN/m²) will remain on the concave surface. A comparison of the data shown in figure 21(a) with that shown in reference 2 for specimens with 25 ksi (170 MN/m²) residual outer-fiber stress and exposed at 600° F (590° K) shows remarkably similar values of relative deflection for given exposure times and indicates that this approximation may be more accurate than might have been expected. Reference 14 also indicates that approximately 30 percent of vibratory treatment compressive stresses are lost by stress relaxation after 1000 hours exposure. This effect would not substantially alter the considerations of the preceding discussion.

The self-stressed specimens were vibrated for longer times after fabrication, approximately 26 hours. Reference 14 indicates that compressive surface stresses due to this treatment are approximately 60 ksi (410 MN/m^2). Algebraically adding this to the 50 ksi (350 MN/m^2) outer-fiber tensile stresses of the self-stressed specimens, a net outer-fiber compressive stress on the order of 10 ksi (70 MN/m^2) should remain. Furthermore, after long exposures at 600° F (590° K) not more than 30 to 40 percent of the compressive stresses should be lost (extrapolation of data from ref. 14) so that the possibility exists that a low tensile stress could exist on the outer fibers of the specimens after exposure. This should be considered a worst case approximation (or a conservative one) because of following reasons: (1) relaxation of the residual tensile stresses in the specimen was not considered, (2) the possibility exists that stress relaxation may have occurred as a percentage of the net compressive stress on the surface, not as a percentage of the compressive stress added by the vibratory treatment. The relative shortening data for exposure times up to 15 000 hours (fig. 21(b)) indicating no sign of stress corrosion certainly appear to bear out this reasoning.

A secondary factor concerning the ability of vibratory cleaning to apply significant magnitudes of compressive stresses to prevent or alleviate hot-salt stress corrosion in titanium-alloy parts may be the difference in configuration between the residual-stress and self-stressed specimens. The outer-fiber tensile stresses in the self-stressed specimens are on convex surfaces which are relatively accessible to the vibrating aluminum oxide triangles during the vibratory-cleaning process. The outer-fiber tensile stresses in the residual-stress specimens are on concave surfaces which may not be quite as accessible to the triangles. It is possible that surfaces in grooves might not be treated at all. The process, however, seems quite feasible and inexpensive for the simultaneous treatment of many small parts without deep grooves.

In summary of the results for vibratory-cleaned specimens, this procedure appears to be effective for preventing or alleviating hot-salt stress corrosion in Ti-8Al-1Mo-1V alloy sheet after at least 15 000 hours exposure at 600° F (590° K), if sufficient vibrating time, at least 26 hours, is given the specimens after fabrication and if the specimens have surfaces accessible to the vibrating triangles. The appearance of stress-corrosion cracks in residual-stress specimens after 6000 and 8000 hours exposure is attributed to insufficient vibratory treatment time. This vibratory treatment is not considered to be applicable to large structural panels but would be a simple and inexpensive method of simultaneous treatment of many small titanium-alloy parts.

Comparison of Coatings and Surface Treatments

The comparative effectiveness of the coatings and surface treatments investigated in this study in prevention of hot-salt stress corrosion in Ti-8Al-1Mo-1V alloy sheet is

shown in figure 26(a) for residual-stress specimens and in figure 26(b) for self-stressed specimens. The curves in this figure were generated from data in previous figures by connecting the lowest relative deflection or relative shortening data points at each exposure time for each coating or surface treatment. Using this method produces conservative data curves but highlights the effects of variability in test specimen behavior. The curves of figures 26(a) and 26(b) are generally descriptive of the behavior of the coating and surface treatment specimens, which were described in previous sections of this report. The effectiveness of the aluminum coating is indicated in this figure. The reduction in ductility of the nickel-coated specimens after 10 000 hours exposure can be noted. The polyimide coating was very effective until its deterioration. The possible variability in peening conditions resulting in reduction in ductility and subsequent increase for the glass-bead-peened specimens is noted for both residual-stress and self-stressed specimens. The effectiveness of the vibratory-cleaning treatment for self-stressed specimens is shown in figure 26(b) but the appearance of stress corrosion in the vibratory-cleaned residual-stress specimens because of insufficient vibratory treatment time is apparent in figure 26(a).

In general, the curves of figure 26 show that the coatings and surface treatments investigated were effective in prevention or alleviation of salt stress corrosion in Ti-8Al-1Mo-1V alloy sheet for long periods of time at 600° F (590° K). The exceptions were the polyimide coating which was effective up to approximately 750 hours and the vibratory treatment which was ineffective after approximately 4000 hours exposure for the residual-stress specimens because of insufficient vibratory treatment time.

CONCLUDING REMARKS

The effectiveness of three coatings and two surface treatments in preventing or alleviating salt stress corrosion of residual-stress and self-stressed Ti-8Al-1Mo-1V (duplex annealed) titanium-alloy sheet was investigated for longtime exposures in air at sea-level atmospheric pressure and 600° F (590° K). Brief conclusions for the coatings are presented in the order of their effectiveness and for the surface treatments in the order of their effectiveness and general applicability.

Coatings

1. The electrophoretically deposited aluminum coating apparently prevented hot-salt stress corrosion for the test duration of 10 000 hours. Substrate ductility was evidently not reduced by the coating process but other possible effects of the sintering treatment on the substrate should be investigated. This process appears applicable to both large and small parts of complex shape.

2. The electroplated nickel coating prevented hot-salt stress corrosion for 10 000 hours exposure. However, reduced substrate ductility of both salt-dipped and control specimens after 10 000 hours exposure indicated the possibility of interface hardening due to solid-state diffusion between coating and substrate. The nickel plating process and "black nickel" overcoat for high emittance are standard commercial processes.

3. The polyimide-resin coating was adherent and ductile as applied to the specimens but embrittled with exposure and peeled off the specimens after 750 to 1000 hours exposure, apparently because of continued polymerization and oxidation effects. The polyimide coating provided good protection against salt stress corrosion up to 500 hours exposure. The coating was applied by a simple dipping process.

Surface Treatments

1. The glass-bead-peening process appeared to be effective in preventing or alleviating hot-salt stress corrosion for at least 10 000 hours at 600⁰ F (590⁰ K). An apparent loss of 30 to 50 percent of ductility in the titanium alloy after 3000 hours exposure which was regained after further exposures is not definitively explained. The glass-bead-peening process is inexpensive and applicable for both small complex shapes and larger areas of a structure.

2. The vibratory-cleaning process appeared to be effective for preventing or alleviating hot-salt stress corrosion for at least 15 000 hours at 600⁰ F if sufficient vibrating time, at least 26 hours, was given to the parts after fabrication. This inexpensive process is primarily applicable to the simultaneous treatment of many small parts with surfaces accessible to the treatment.

Langley Research Center,

National Aeronautics and Space Administration,

Langley Station, Hampton, Va., July 14, 1967,

129-03-06-06-23.

APPENDIX A

CONVERSION OF U.S. CUSTOMARY UNITS TO SI UNITS

The International System of Units (SI) was adopted by the Eleventh General Conference on Weights and Measures held in Paris, October 1960, in Resolution No. 12 (ref. 8). Conversion factors required for units used herein are given in the following table:

Physical quantity	U.S. Customary Unit	Conversion factor (*)	SI Unit
Length	inches (in.)	2.54	centimeters (cm)
Pressure	torr	133.32	newtons per square meter (N/m ²)
Stress	kips per square inch (ksi)	6.896	meganewtons per square meter (MN/m ²)
Temperature	(°F + 459.67)	5/9	degrees Kelvin (°K)

*Multiply value given in U.S. Customary Unit by conversion factor to obtain equivalent value in SI Unit.

Prefixes to indicate multiples of units are as follows:

Prefix	Multiple
mega (M)	10 ⁶
kilo (k)	10 ³
centi (c)	10 ⁻²
milli (m)	10 ⁻³
micro (μ)	10 ⁻⁶

APPENDIX B

COATING AND SURFACE TREATMENT APPLICATION PROCEDURES

The coatings and surface treatments used in this study were selected so that commercially feasible processes, which could be applied relatively inexpensively to large fabricated parts, would be investigated. The details of coating or surface treatment application procedures are given in the following paragraphs.

Coatings

Nickel coating.- Nickel coatings were applied to the specimens by a conventional electroplating process which is widely used in industry for nickel plating of steels. The process was controlled to produce a coating thickness of approximately 0.0005 inch (13 μm). The coating procedure was as follows:

1. Degrease specimens in trichloroethylene
2. Alkaline clean in "Vitroklene" and rinse in tap water
3. Pickle until bright in a solution of 12 parts nitric acid, 3 parts hydrofluoric acid, 128 parts water. Rinse in distilled water at room temperature.
4. Nickel plate in sulfamate bath. Follow with a quick distilled water rinse at room temperature.

Details of the sulfamate coating bath and other parameters of the process are given in reference 15.

5. Apply a thin "black nickel" overcoat in a bath containing 10 parts (by weight) nickel chloride, 4 parts ammonium chloride, 2 parts sodium thiocyanate, 4 parts zinc chloride, and 120 parts water. The bath is maintained at a pH of 5.0, at room temperature, and the overcoat is applied at a current density of 1.5 amps/ft² (16 A/m²).

6. Rinse in tap water and air dry.

The black nickel overcoat was applied to the specimens because of the low emittance of the sulfamate nickel plating. Application of the overcoat raised the specimen emittance from 0.09 to 0.97. Emittance values for uncoated Ti-8Al-1Mo-1V and for other coatings are given in appendix C.

Aluminum coating.- The aluminum coatings were applied to the specimens by electrophoretic deposition. Electrophoresis was utilized to provide coatings of more uniform thickness than those attainable by hot dipping. This electrophoretic method is especially advantageous if complex parts are to be coated. The coating equipment consisted of a stainless-steel tank through which a dispersion of the coating material was

APPENDIX B

continuously pumped. The tank served as a positive electrode while the specimen to be coated was connected to the negative electrode of a dc power supply.

The aluminum-coating dispersion was supplied by Vitro Laboratories. It was produced in the following manner: aluminum powder was ball milled with a protein-like additive. This procedure produced a powder containing particles approximately 10 microns ($10\ \mu\text{m}$) in diameter with a positive static charge on the surface. The dispersion fluid contained 60 percent (by volume) nitromethane and 40 percent isopropanol. To this fluid, 2 percent (by weight) of the milled aluminum powder was added. The coating was electrophoretically applied at 150 volts for 15 seconds to specimens which had been washed in acetone after chemical cleaning. The specimens were air dried.

In order to obtain high-density coatings which are well bonded to the substrate, many electrophoretic coatings must be isostatically cold pressed and subsequently sintered or hot pressed. However, it was found in this investigation that liquid phase sintering at 1200°F (920°K) for 15 minutes in a vacuum of 10^{-4} to 10^{-5} torr (10 to $1\ \text{MN/m}^2$) provided a well bonded uniform coating approximately 0.001 inch ($25\ \mu\text{m}$) thick on all surfaces of the titanium-alloy specimens with no need for isostatic pressing. Elimination of the isostatic pressing cycle makes this process useful for coating of large, complex titanium-alloy parts. The sintering cycle involved in this coating process resulted in some stress relief in the self-stressed specimens. Reference 16 indicates a relaxation of 80 percent of the stresses in mill annealed sheet for conditions similar to the sintering process. A similar degree of stress relief was noted for the self-stressed specimens of this investigation in X-ray measurements of stress by the methods described in reference 14. X-ray stress measurements could not be made on residual-stress specimens because of their sharp curvature. However, the residual-stress specimens of this investigation probably retained significant tensile stresses on the concave surface after sintering, since reference 13 indicates that no alleviation of salt-stress-corrosion cracking was noted after vacuum heating for 1 hour at 1200°F (920°K). It is therefore believed that the effectiveness of the aluminum coating in preventing salt stress corrosion for residual-stress specimens was not simply due to a stress relief, but to the existence of the aluminum outer layer on the specimen surface.

Polyimide coating.- A commercial polyimide-resin wire enamel, designated R.C. 5057, from E. I. duPont deNemours, Inc., was used to coat some titanium-alloy specimens by a hot-dipping procedure, as follows:

1. Heat chemically cleaned specimens to 300°F (420°K) in air.

2. Dip hot specimen in room-temperature polyimide resin which has been thinned with dimethyl acetamide or xylene to a concentration of 10- to 12-percent polymer solids by weight.

APPENDIX B

3. Rapidly remove specimen from resin, allow excess to run off, then heat in air at 300° F (420 K) for 1 hour to evaporate all solvent.

4. Invert specimen and repeat steps 2 and 3. Air cool.

5. Insert specimens into an air oven at 700° F (640° K) and bake for 5 minutes. After this final polymerizing bake, remove specimens from oven and air cool.

This procedure resulted in a well bonded smooth coating, approximately 0.0005 inch (13 μm) thick.

Surface Treatments

It is generally recognized that a tensile stress on the outer fiber of a stressed part must be present before stress corrosion will occur. Any surface treatments which will effectively remove the tensile stress or induce a compressive stress in the outer fiber should be effective in alleviating or preventing stress corrosion. A process such as peening or vibratory cleaning would have this effect and would have the advantage of not adding the weight of a coating to the titanium-alloy sheet, but any deleterious effects such as a change in mechanical properties of the material must be minimized. Moreover, the possibility that the desirable compressive stresses could be lost by stress relaxation after long exposure times at elevated temperatures must be investigated before these surface treatments can be used with confidence.

Glass-bead peening.- Preliminary results of the effect of glass-bead peening on residually stressed specimens were presented in reference 13. That study showed that peening with glass particles was promising for alleviating stress corrosion. Reference 13 also showed that peening with coarse glass particles reduced ductility for as-fabricated specimens by a considerable amount, but that specimens peened with fine glass particles did not exhibit this undesirable characteristic. Accordingly, specimens for the present study were all peened with fine glass particles, 44 to 74 microns (44 to 74 μm) in diameter. The chemically cleaned residual-stress specimens were peened at 60 psi (410 kN/m^2) air pressure for at least 15 seconds on all surfaces. The self-stressed specimens were peened under the same conditions but only on the convex surfaces.

Vibratory cleaning.- The vibratory-cleaning process was originally used to eliminate hand deburring in specimen preparation. It was found that this process also alleviated salt stress corrosion. The treatment consisted of immersion of the specimens in a tank filled with aluminum oxide triangles (0.25 inch (6.4 mm) thick by 0.62 inch (16 mm) on a side), detergent, and water. The box was vibrated at about 30 cycles per second (30 Hz), which was sufficient to impart a rolling motion to the entire mass. Burrs were removed, all edges were slightly rounded, and the visual appearance of the flat surfaces of the specimens was different from that of as-rolled Ti-8Al-1Mo-1V sheet.

APPENDIX B

However, a surface-roughness determination indicated approximately the same roughness for vibrated and as-rolled sheet.

The two different types of specimens were subjected to this treatment in different ways. The residual-stress specimens were vibrated for 4 hours after forming. The self-stressed specimens were vibrated for 6 hours as a deburring treatment to remove burrs from the shearing process. The strips were then machined to the appropriate dimensions and then the strips were subjected to an additional 20-hour vibratory treatment to remove the burrs from the machining process. The specimens were then fabricated (load-induced stresses applied).

APPENDIX C

EMITTANCE OF COATED Ti-8Al-1Mo-1V SHEET

Alleviation of aerodynamic heating effects on structures and systems is one of the main considerations in design, fabrication, and operation of a Mach 2.7 supersonic transport. Even relatively small decreases in aircraft skin equilibrium temperature during cruising flight may result in significant reduction in cooling-system capacity requirements. Higher values of skin thermal emittance will result in lower skin equilibrium temperatures through increased thermal radiation from the skin to the atmosphere, if other factors are equal. Titanium-alloy surfaces as well as those of most other metals exhibit low-emittance values; thus a high-emittance coating may be required. Two of the coatings investigated in this study for prevention of salt stress corrosion were subjected to emittance measurement tests.

A double-beam spectrophotometer was used to measure reflectance of uncoated and coated titanium alloys throughout the wavelength range of 2.5 μm to 15 μm at small wavelength intervals. From these reflectance data, total normal emittance values were calculated. Details of the equipment and procedures used are given in reference 15. The specimens were small disks, 0.9 inch (2.3 cm) diameter by approximately 0.050 inch (1.3 mm) thick. All specimens were maintained at 75° F to 80° F (297° K to 300° K).

Total normal emittance values for uncoated and coated Ti-8Al-1Mo-1V alloy sheet are given in the following table:

Specimen	Emittance
Uncoated sheet, as rolled	0.17
Uncoated sheet, chemically cleaned	.18
Nickel plating, no overcoat	.09
Nickel plating with "black nickel" overcoat	.97
Polyimide-resin coating	.69

The uncoated sheet, both in the as-rolled and chemically cleaned conditions, exhibited low-emittance values typical of most metal surfaces. The nickel plating without the overcoat lowered emittance even further. The addition of the "black nickel" overcoat to the nickel plating raised emittance to a very high value, 0.97, which approaches the maximum possible emittance, 1.0 (a black body). The increase in emittance afforded by the black nickel overcoat could measurably lower the equilibrium temperature of an aerodynamically heated surface. Although the polyimide-resin coating was transparent, that is, the titanium-alloy surface was visible through it, a significant emittance increase was provided by it as compared with the uncoated titanium alloy.

REFERENCES

1. Braski, David N.; and Heimerl, George J.: The Relative Susceptibility of Four Commercial Titanium Alloys to Salt Stress Corrosion at 550° F. NASA TN D-2011, 1963.
2. Dexter, Howard B.: Salt Stress Corrosion of Residually Stressed Ti-8Al-1Mo-1V Alloy Sheet After Exposure at Elevated Temperatures. NASA TN D-3299, 1966.
3. Kochka, E. L.; and Petersen, V. C.: The Salt Corrosion of Titanium Alloys at Elevated Temperatures. Final Tech. Rept. (Contract No. NOas 60-6004-c), Crucible Steel Co. of Am., Jan. 15, 1961.
4. Petersen, V. C.; and Bomberger, H. B.: The Mechanism of Salt Attack on Titanium Alloys. Stress-Corrosion Cracking of Titanium, Spec. Tech. Publ. No. 397, Am. Soc. Testing Mater., c.1966, pp. 80-94.
5. Covington, L. C.; and Early, F. R.: Methods of Protecting Titanium Against Hot Salt Stress Corrosion. Progr. Rept. No. 21, Titanium Metals Corp. of Am., Aug. 1964.
6. Braski, David N.: Preliminary Investigation of Effect of Environmental Factors on Salt Stress Corrosion Cracking of Ti-8Al-1Mo-1V at Elevated Temperatures. NASA TM X-1048, 1964.
7. Honeycutt, J. O., Jr.; and Willhelm, A. Clyde: Effects of Commercially Available Protective Coatings on Stress-Corrosion Properties of Supersonic-Transport Skin Materials. Contract No. NASr 117, Southern Res. Inst., Mar. 22, 1966.
8. Mechtly, E. A.: The International System of Units - Physical Constants and Conversion Factors. NASA SP-7012, 1964.
9. Heimerl, G. J.; Braski, D. N.; Royster, D. M.; and Dexter, H. B.: Salt Stress Corrosion of Ti-8Al-1Mo-1V Alloy Sheet at Elevated Temperatures. Stress-Corrosion Cracking of Titanium, Spec. Tech. Publ. No. 397, Am. Soc. Testing Mater., c.1966, pp. 194-214.
10. Todd, N. W.; and Wolff, F. A.: Polyimide Plastics Withstand High Temperatures. Materials in Design Engineering, vol. 60, no. 2, Aug. 1964, pp. 86-91.
11. Stein, Bland A.; and Pride, Richard A.: Effects of 450° F and 600° F Exposures on the Mechanical Properties of Polyimide/Glass-Fiber Honeycomb Sandwiches and Laminated Beams. AIAA Paper No. 67-174, Jan. 1967.
12. Teichman, Louis A.; and Burks, Harold D.: An Automatic System for Determining Solar Absorptance and Thermal Emittance of Surfaces from Spectral Normal Reflectance Measurements. NASA TM X-1113, 1965.

13. Pride, Richard A.; and Woodard, John M.: Salt-Stress-Corrosion Cracking of Residually Stressed Ti-8Al-1Mo-1V Brake-Formed Sheet at 550° F (561° K). NASA TM X-1082, 1965.
14. Braski, David N.; and Royster, Dick M.: X-Ray Measurement of Residual Stresses in Titanium Alloy Sheet. Advances in X-Ray Analysis, Vol. 10. Proceedings of the Fifteenth Conference J. B. Newkirk, and G. R. Mallett, eds., Plenum Press, Inc., 1967.
15. Lyman, T., ed.: Metals Handbook. Volume 2.- Heat Treating, Cleaning and Finishing. 8th ed., Am. Soc. Metals, c.1964.
16. Wood, R. A.: The Ti-8Al-1Mo-1V Alloy. DMIC Rept. S-10, Battelle Mem. Inst., Apr. 1, 1965.

SELF-STRESSED SPECIMENS

RESIDUAL-STRESS SPECIMENS

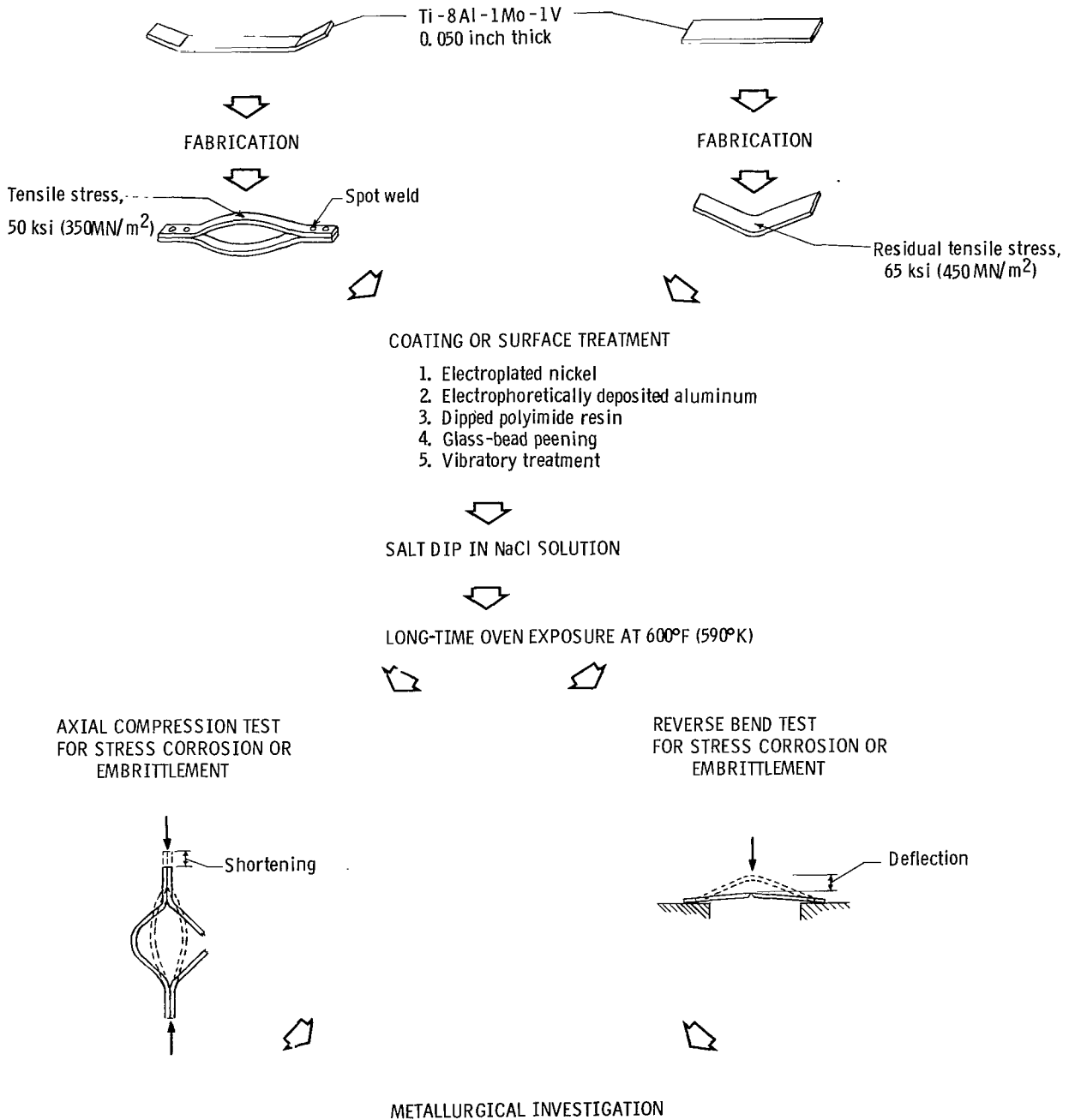


Figure 1.- Outline of experimental study of coatings and surface treatments for prevention of hot-salt stress corrosion in Ti-8Al-1Mo-1V titanium-alloy sheet.

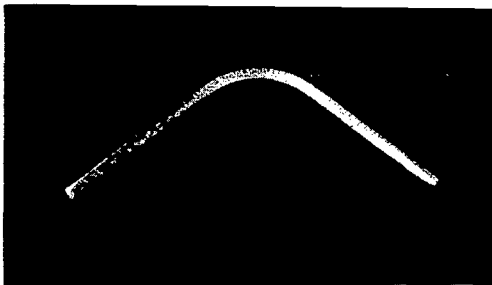


Before

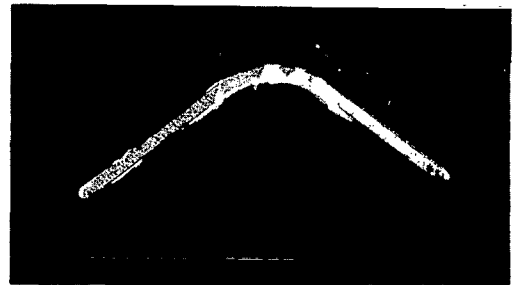


After

(a) Self-stressed specimen.



Before

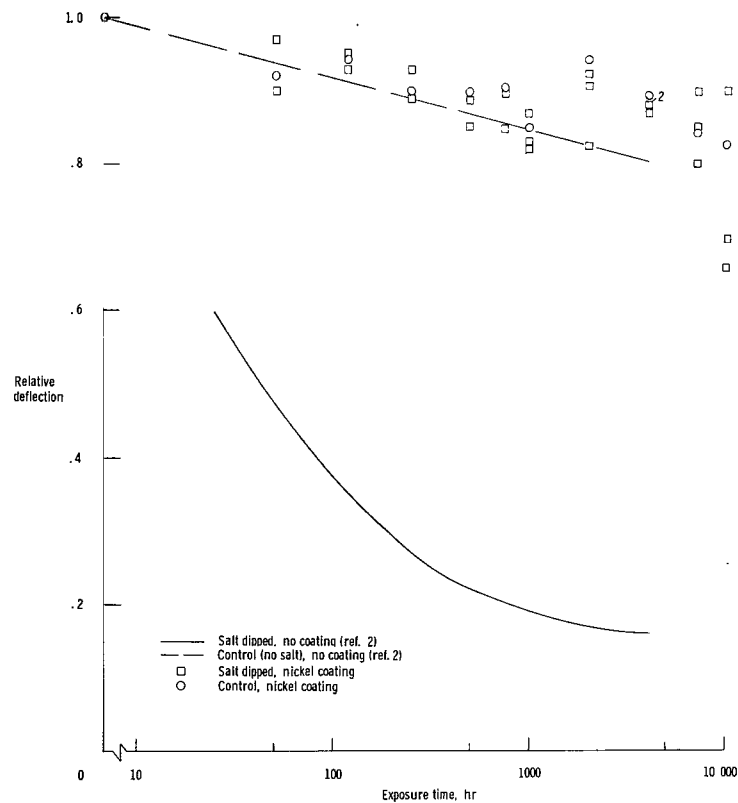


After

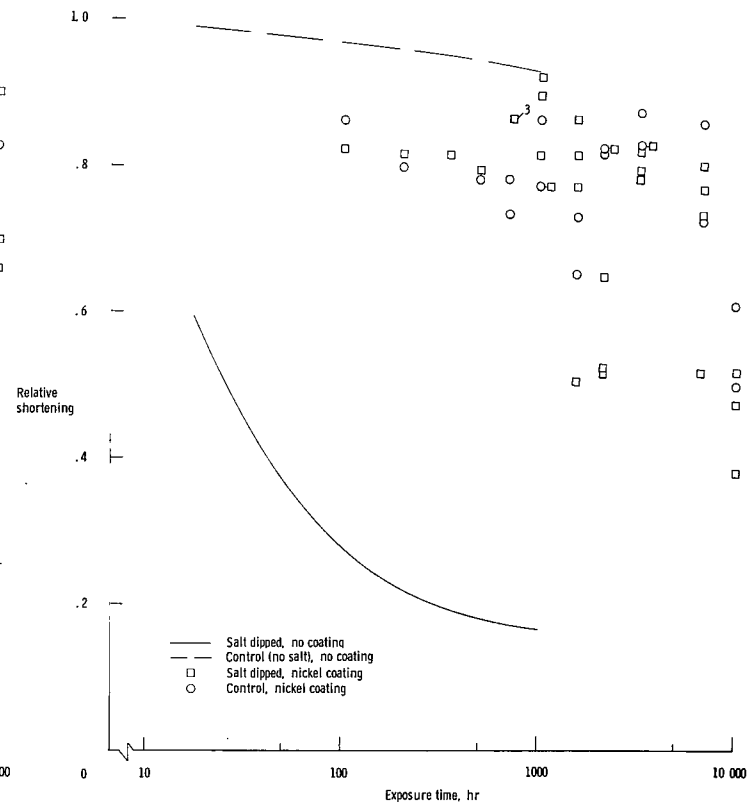
(b) Residual-stress specimen.

L-67-6674

Figure 2.- Self-stress and residual-stress specimens before and after salt dip.

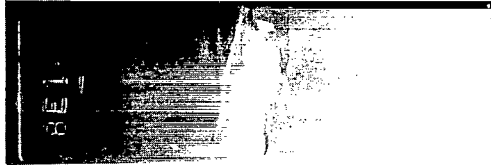


(a) Residual-stress specimens.

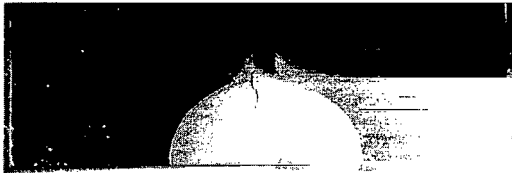


(b) Self-stressed specimens.

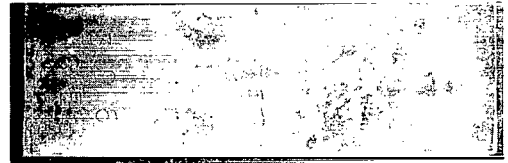
Figure 3.- Effect of nickel coating on the prevention of salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet specimens after 600° F (590° K) exposure.



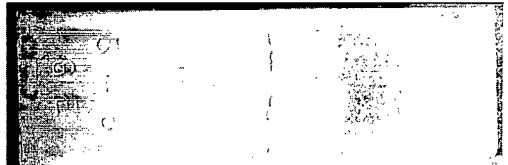
0 hours (no salt)



2000 hours



4000 hours



10 000 hours

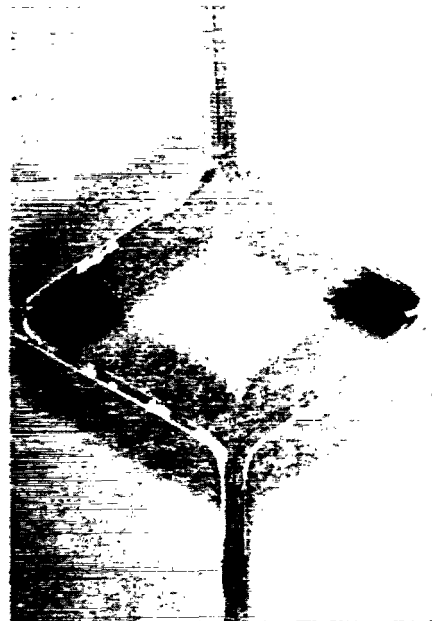
Control

Salt dip

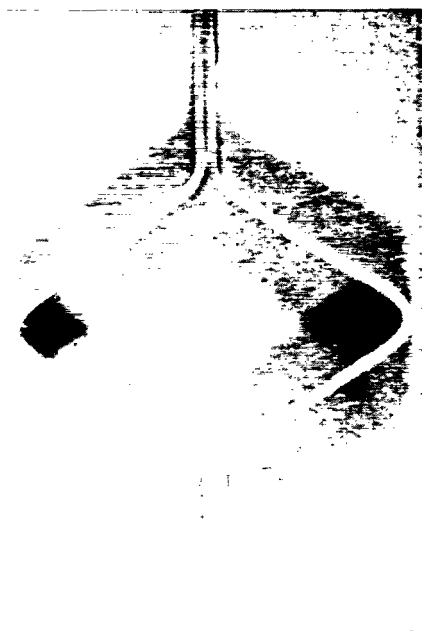
Figure 4.- Nickel-coated residual-stress specimens after various exposures at 600° F (590° K), followed by room-temperature reverse-bend tests. X 2. L-67-6675



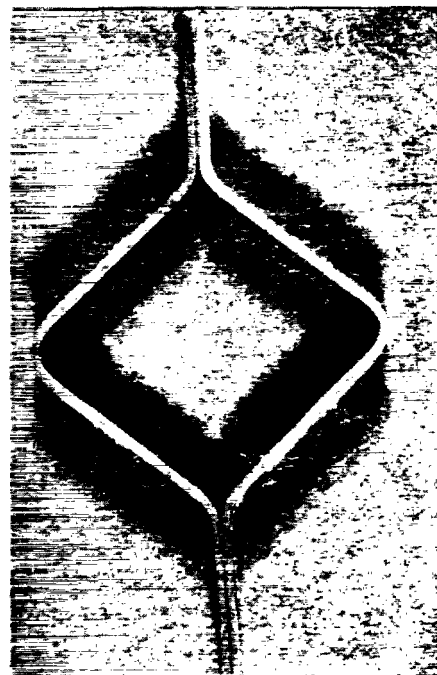
0 hours (no salt)



1000 hours



3200 hours



10 000 hours

Figure 5.- Nickel-coated self-stressed specimens after salt dip and various exposures at 600° F, as noted, followed by room-temperature compression test. L-67-6676

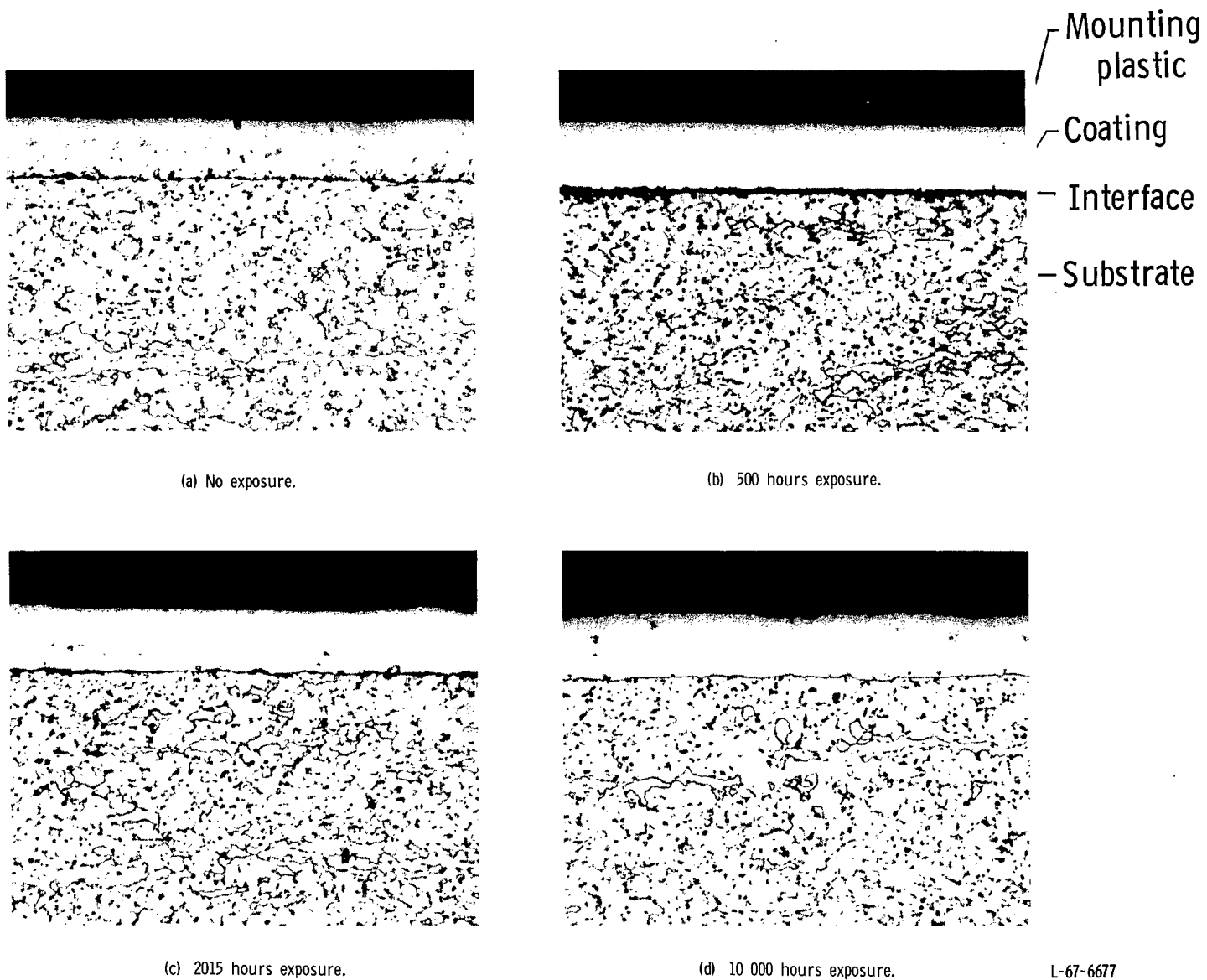


Figure 6.- Cross sections of nickel-coated residually stressed specimens after various 600° F (590° K) exposures and room-temperature mechanical tests. X 500.

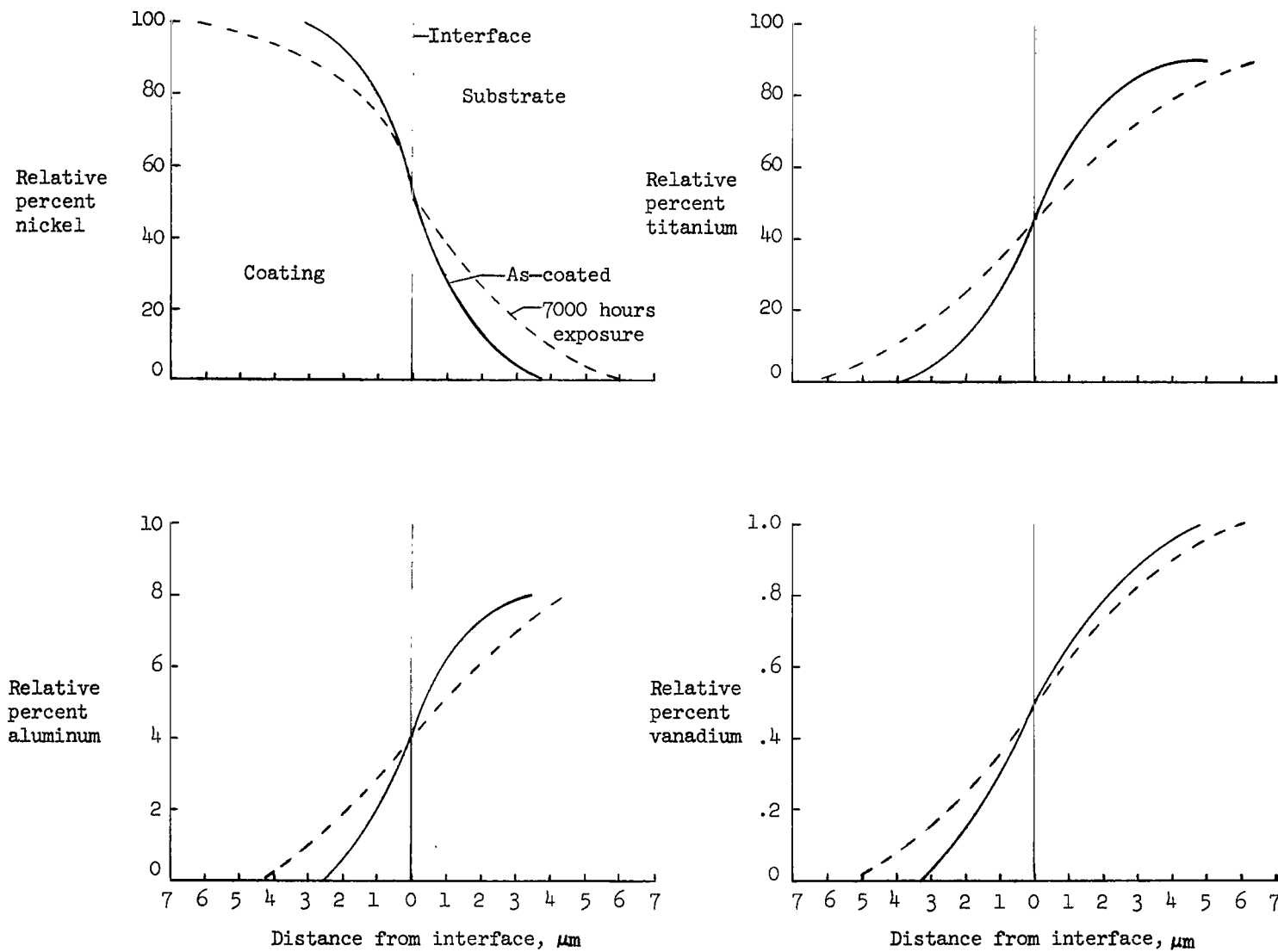


Figure 7.- Results of electron probe microanalyzer scans across substrate-coating interfaces of nickel-coated residual-stress specimens.

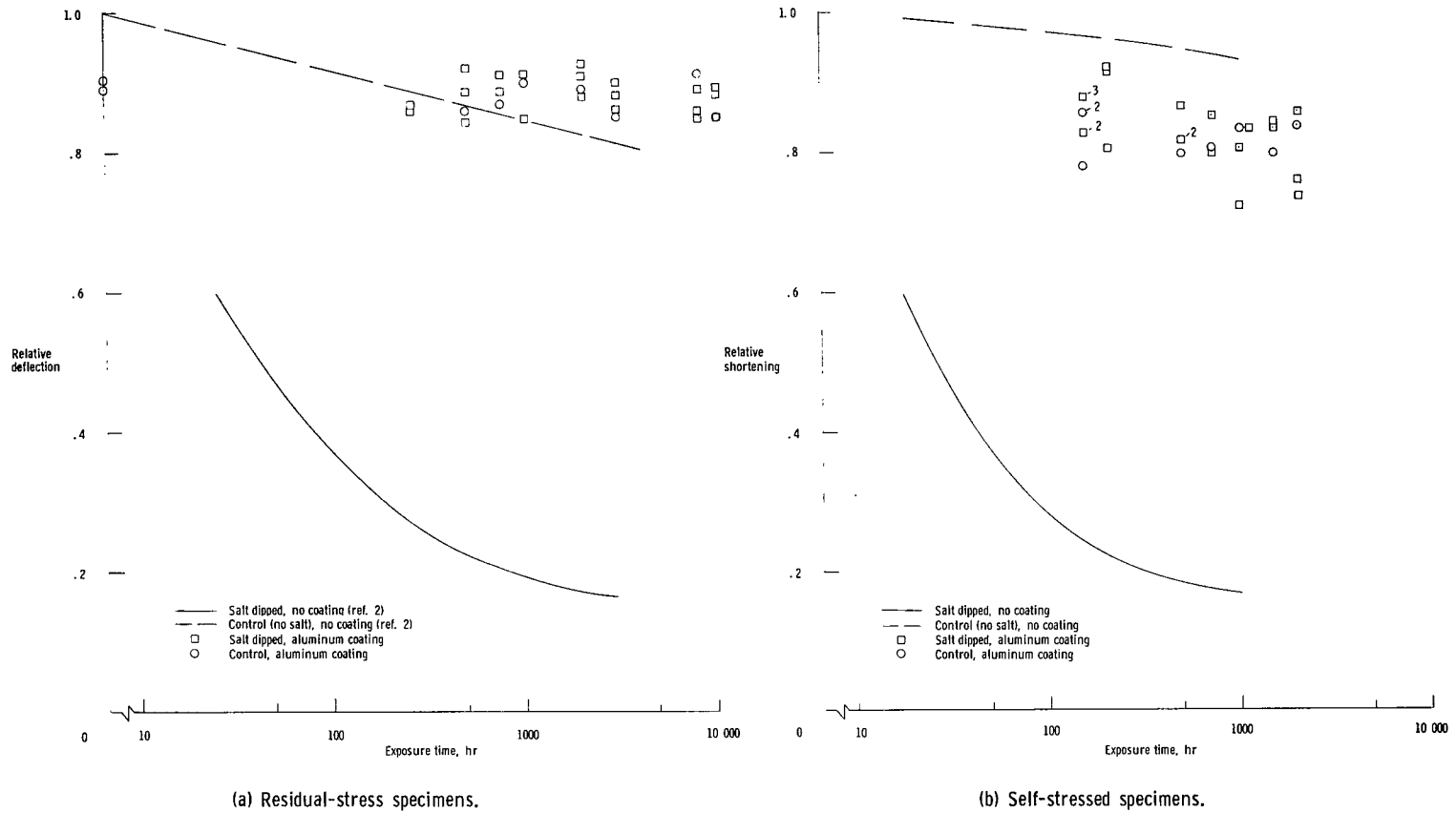
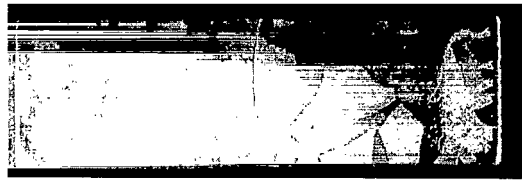
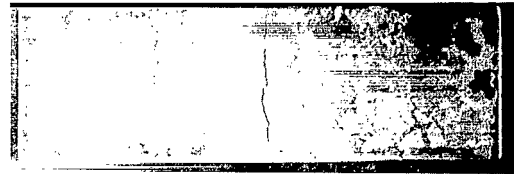
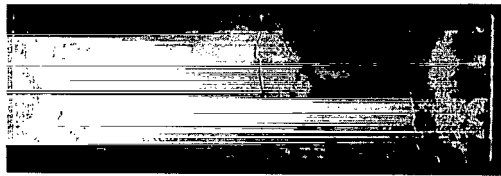


Figure 8.- Effect of aluminum coating on the prevention of salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet specimens after 600° F (590° K) exposure.



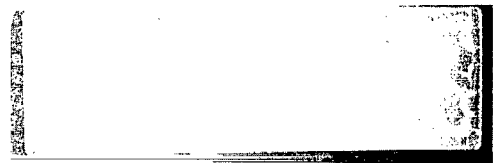
0 hours (no salt)



1000 hours



8000 hours

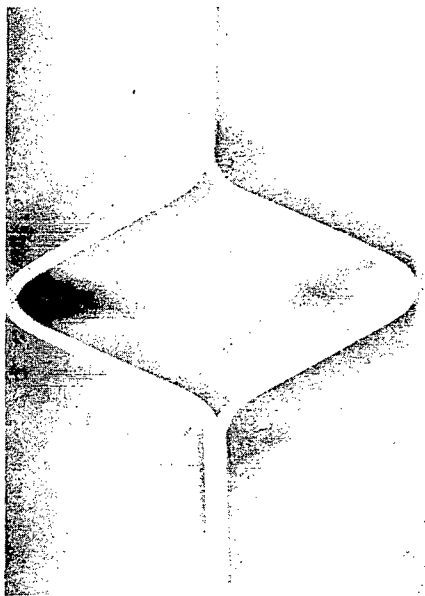


10 000 hours

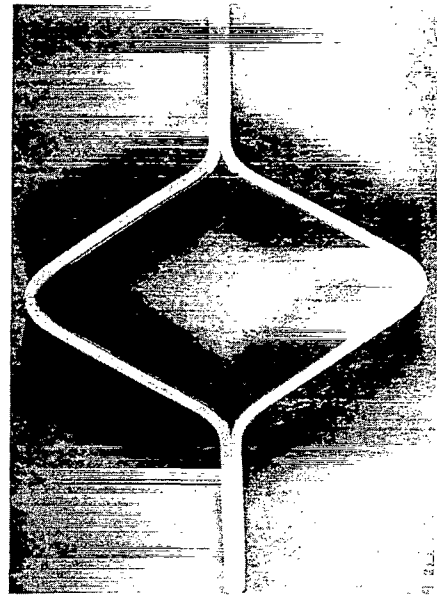
Control

Salt dip

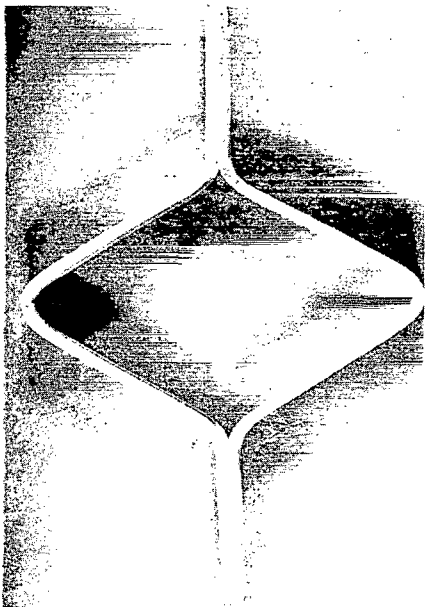
Figure 9.- Aluminum-coated residual-stress specimens after various exposures at 600° F (590° K) followed by room-temperature reverse-bend tests. X 2. L-67-6678



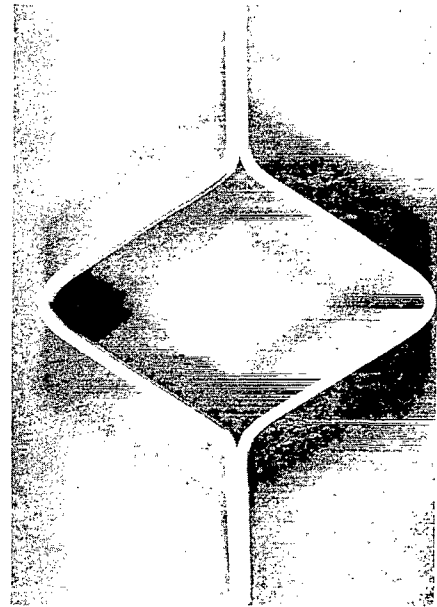
0 hours (no salt)



500 hours

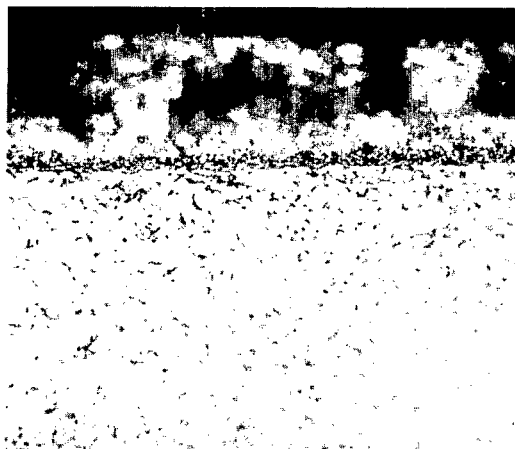


1000 hours

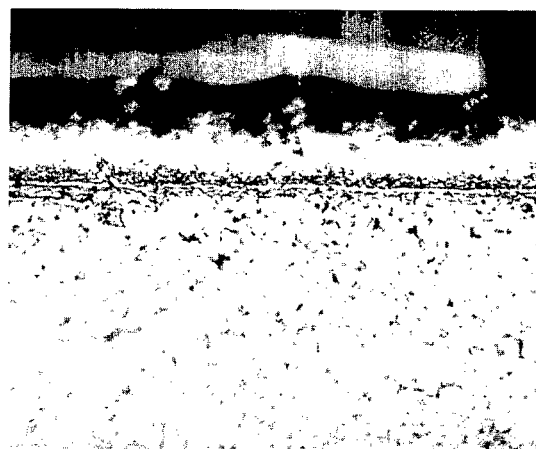


2000 hours

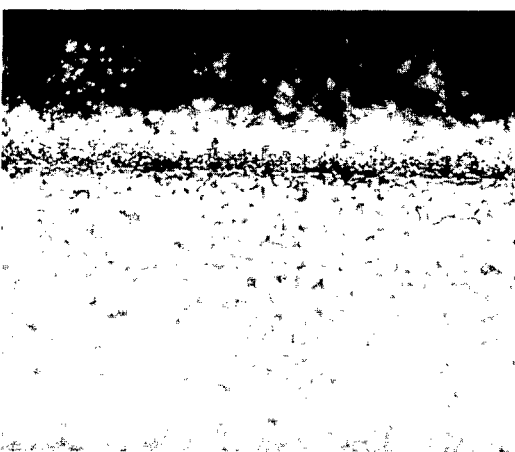
Figure 10.- Aluminum-coated self-stressed specimens after salt dip and various exposures at 600° F (590° K), as noted, followed by room-temperature compression test. L-67-6679



(a) No exposure.



(b) 1000 hours exposure.



(c) 2000 hours exposure.



(d) 10 000 hours exposure.

Mounting plastic

Coating

Interface

Substrate

Light shadowing effect

L-67-6680

Figure 11.- Cross sections of aluminum-coated residually stressed specimens after various 600° F (590° K) exposures and room-temperature mechanical tests. X 500.

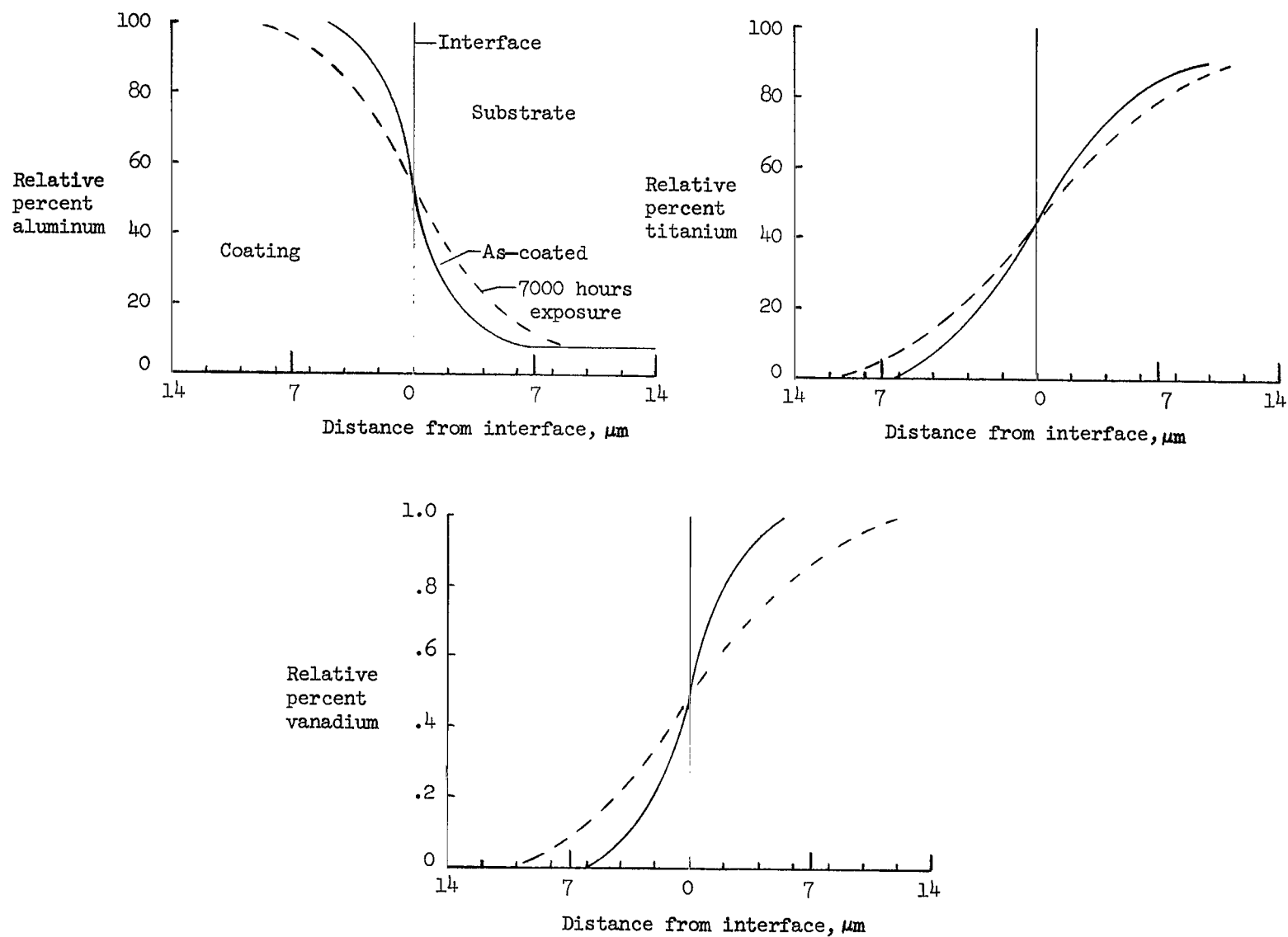
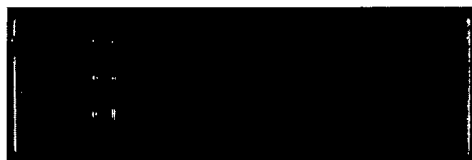


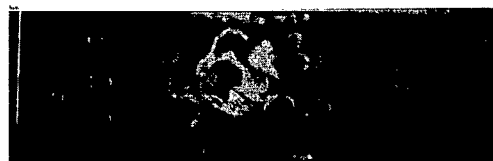
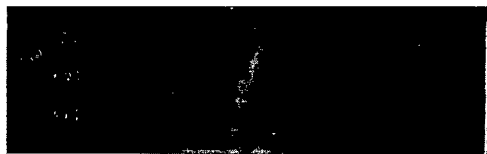
Figure 12.- Results of electron probe microanalyzer scans across substrate-coating interfaces of aluminum-coated residual-stress specimens.



0 hours (no salt)



210 hours



500 hours

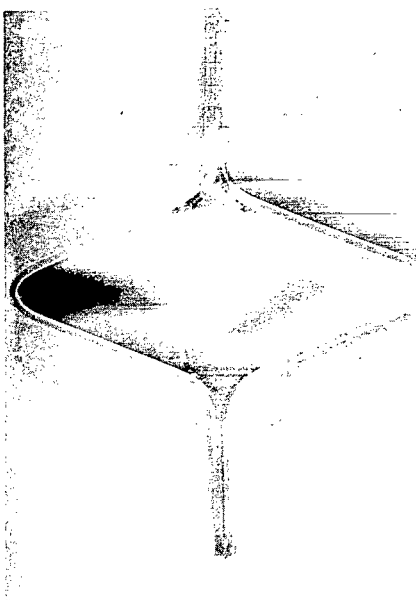


1000 hours

Control

Salt dip

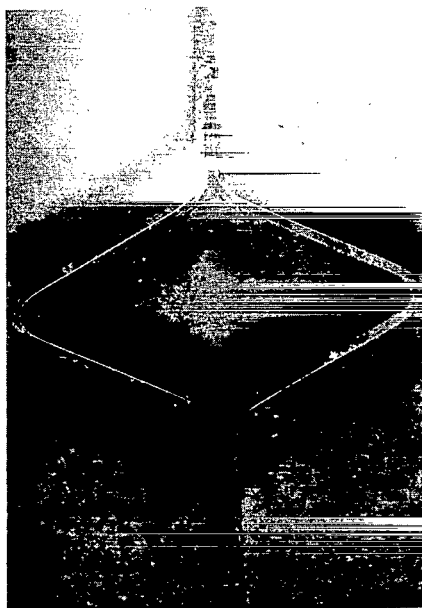
Figure 13.- Polyimide-coated residual-stress specimens after various exposures at 600° F (590° K) followed by room-temperature reverse-bend tests. X 2. L-67-6681



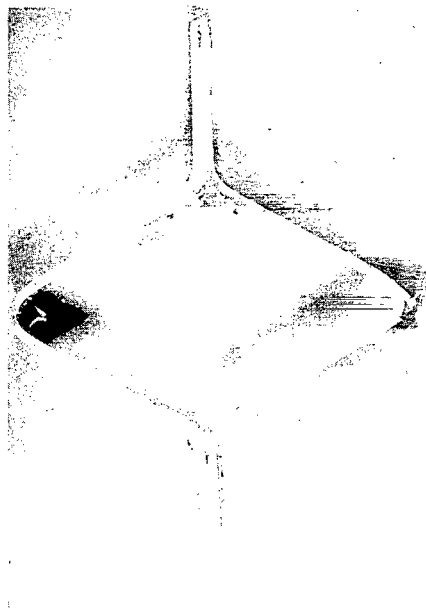
0 hours (no salt)



200 hours

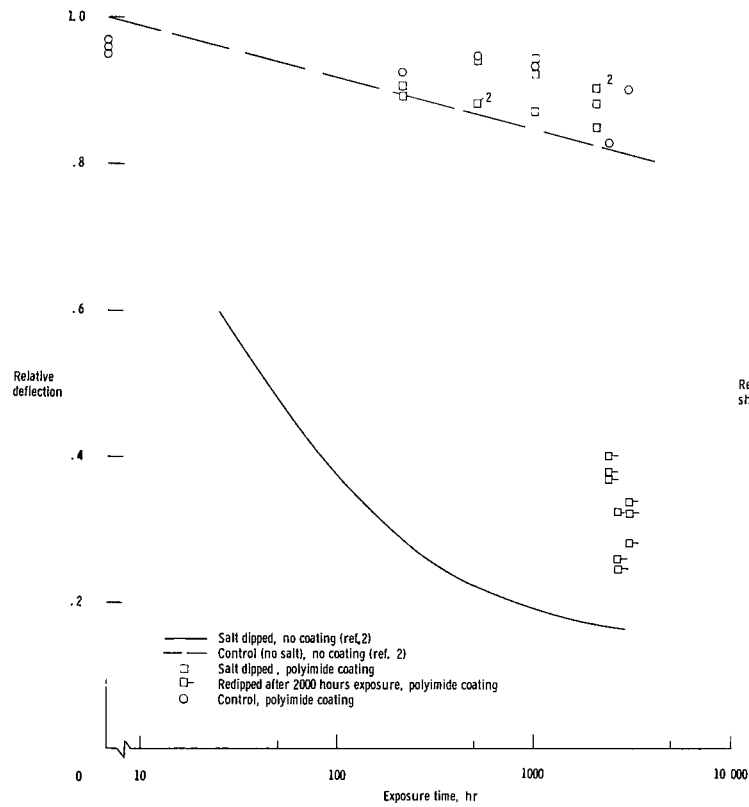


500 hours

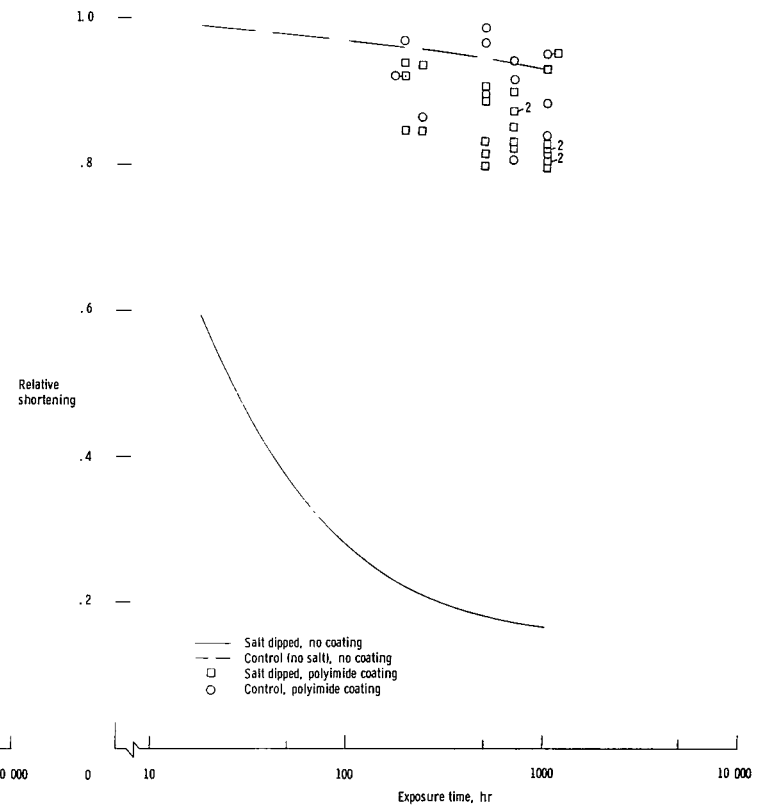


1000 hours

Figure 14.- Polyimide-coated self-stressed specimens after salt dip and various exposures at 600° F (590° K), as noted, followed by room-temperature compression test. L-67-6682

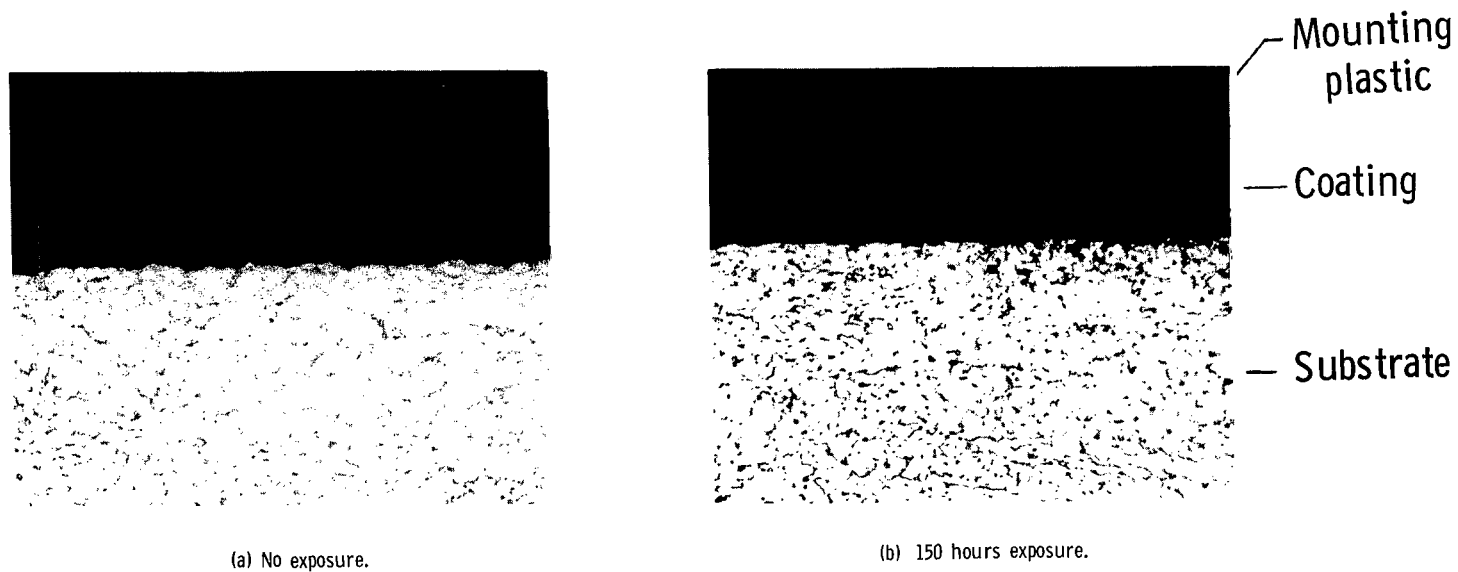


(a) Residual-stress specimens.



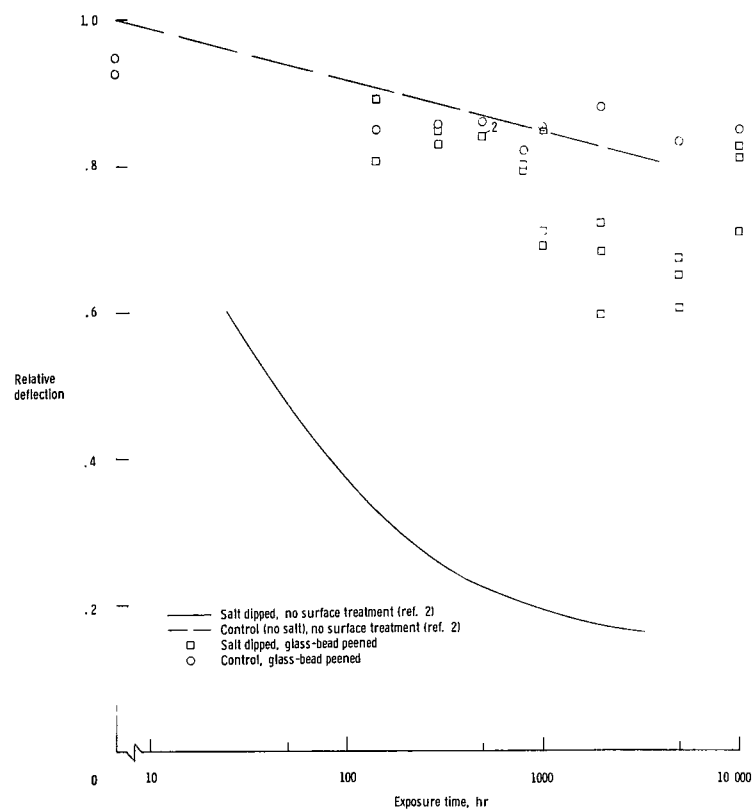
(b) Self-stressed specimens.

Figure 15.- Effect of polyimide coating on the prevention of salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet specimens after 600° F (590° K) exposure.

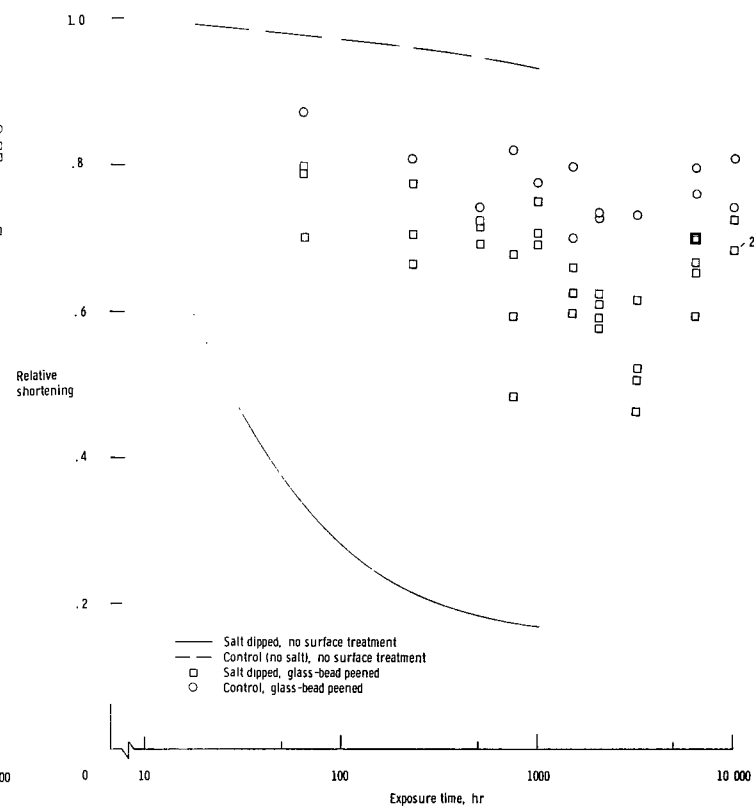


L-67-6683

Figure 16.- Cross sections of polyimide-resin-coated residually stressed specimens after various 600° F (590° K) exposures and room-temperature mechanical tests. X 500.



(a) Residual-stress specimens.

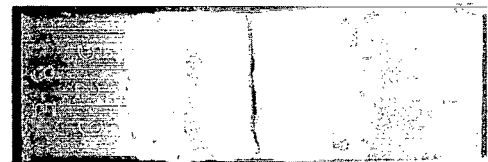
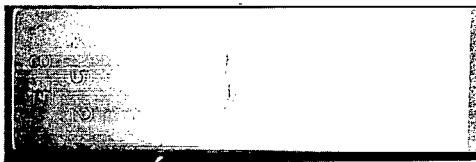


(b) Self-stressed specimens.

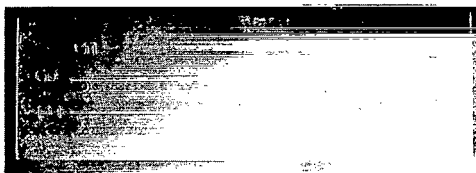
Figure 17.- Effect of glass-bead peening on the prevention of salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet specimens after 600° F (590° K) exposure.



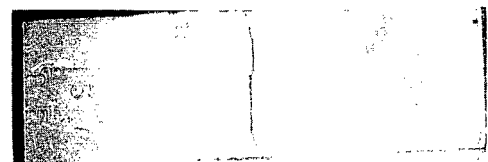
0 hours (no salt)



1000 hours.



5000 hours

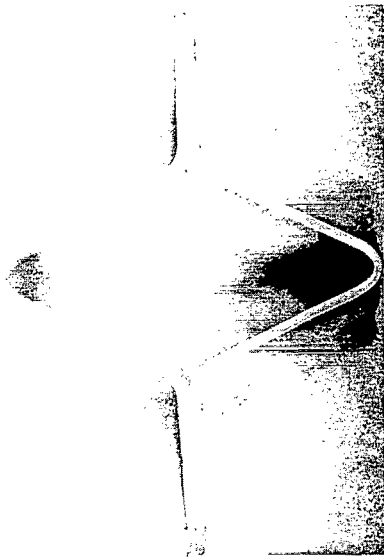


10 000 hours.

Control

Salt dip

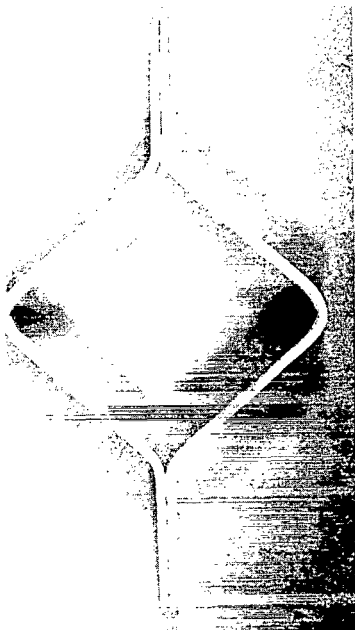
Figure 18.- Glass-bead-peened residual-stress specimens after various exposures at 600° F (590° K) followed by room-temperature reverse-bend tests. X 2. L-67-6684



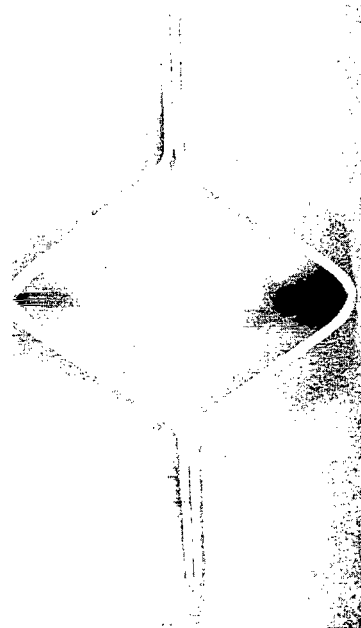
0 hours (no salt)



2000 hours



6400 hours

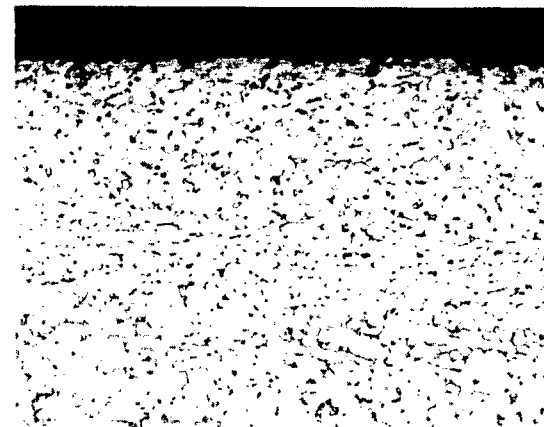


10 000 hours

Figure 19.- Glass-bead-peened self-stressed specimens after salt dip and various exposures at 600° F (590° K), as noted, followed by room-temperature compression test. L-67-6685



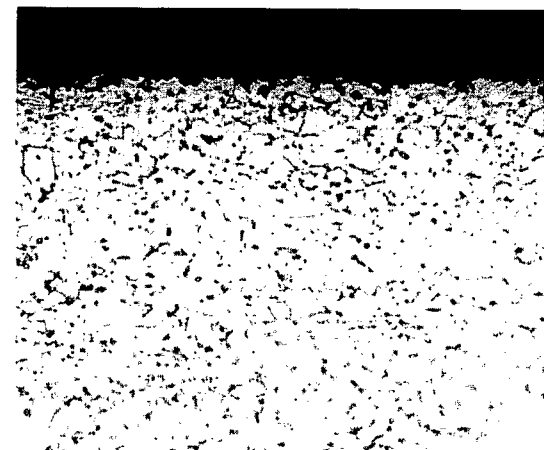
(a) No exposure.



(b) 1000 hours exposure.



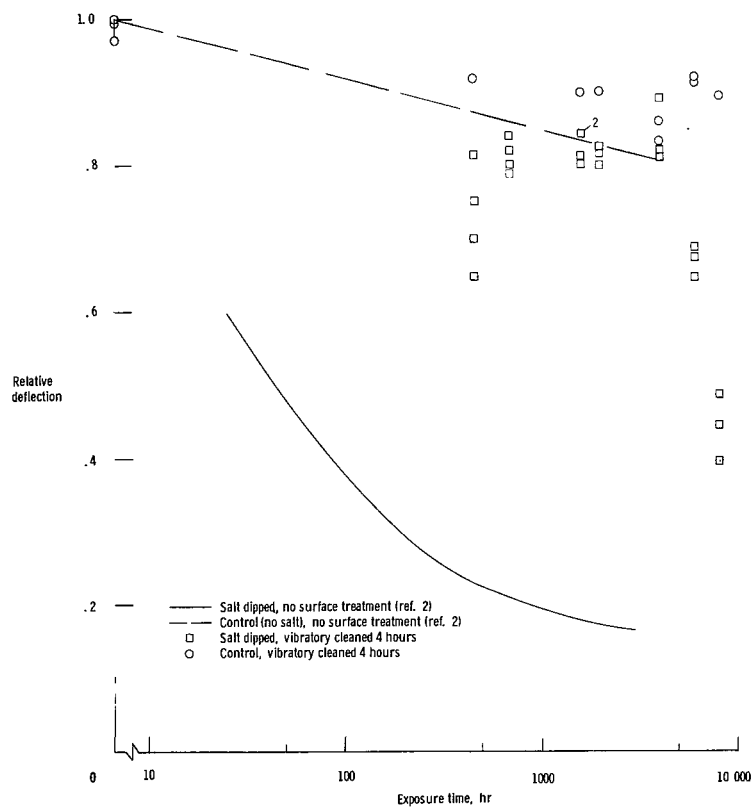
(c) 1992 hours exposure.



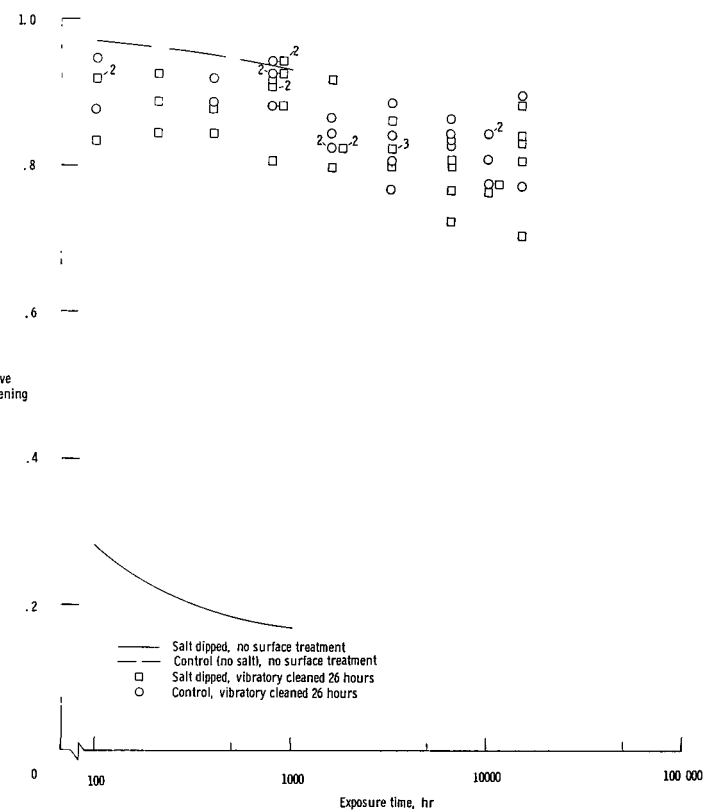
(d) 10 000 hours exposure.

L-67-6686

Figure 20.- Cross sections of glass-bead-peened residually stressed specimens after various 600° F (590° K) exposures and room-temperature mechanical tests. X 500.



(a) Residual-stress specimens.



(b) Self-stressed specimens.

Figure 21.- Effect of vibratory treatment on the prevention of salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet specimens after 600° F (590° K) exposure.

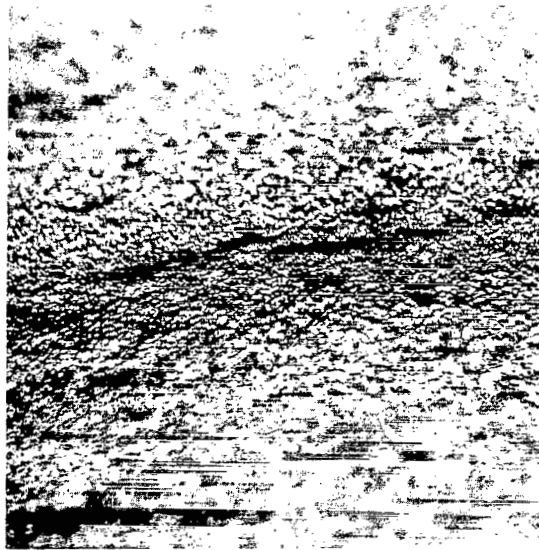
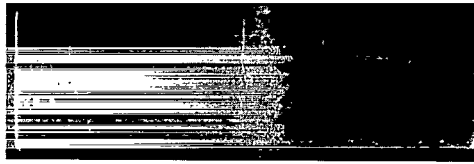
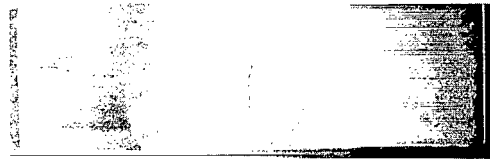
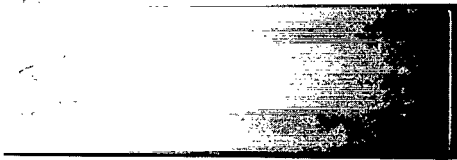


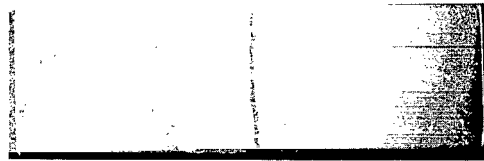
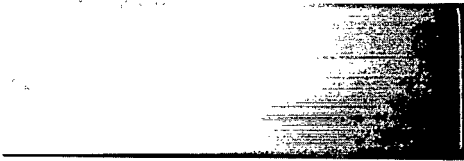
Figure 22.- Surface of vibratory-treated residual-stress specimen after 8000 hours exposure at 600° F (590° K) and room-temperature mechanical test followed by special etch to reveal stress-corrosion cracks. X 75. L-67-6687



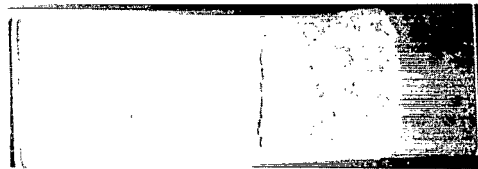
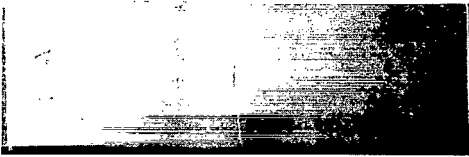
0 hours (no salt)



2000 hours



6000 hours

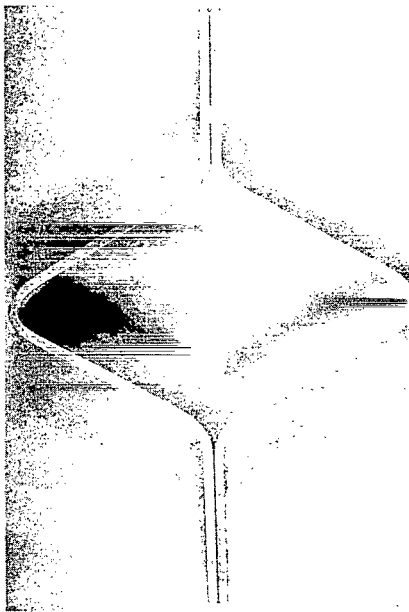


8000 hours

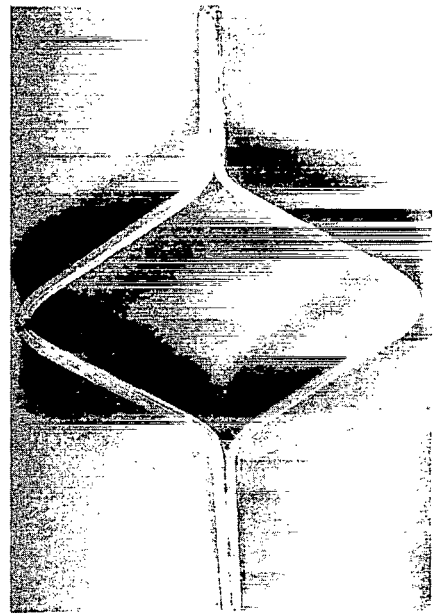
Control

Salt dip

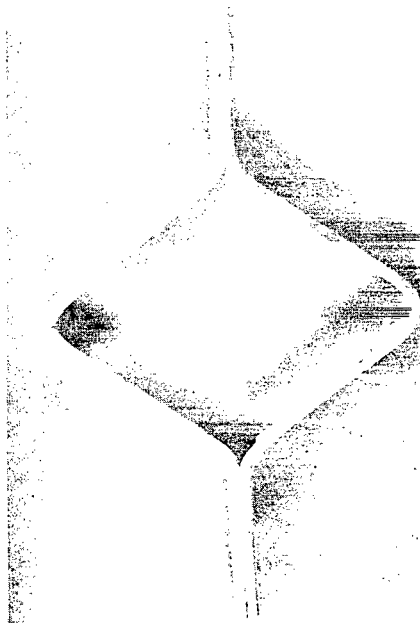
Figure 23.- Vibratory-treated residual-stress specimens after various exposures at 600° F (590° K) followed by room-temperature reverse-bend tests. X 2. L-67-6688



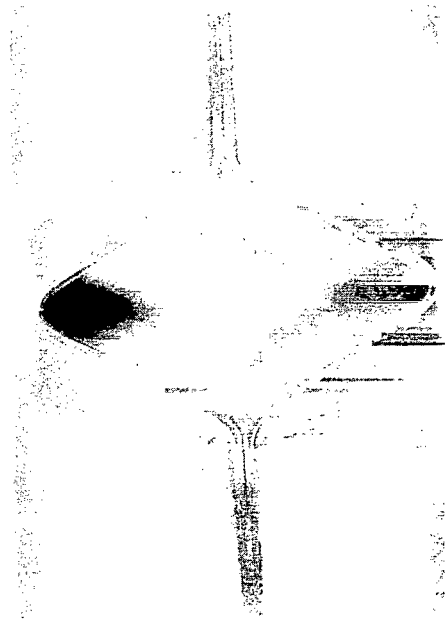
0 hours (no salt)



1000 hours

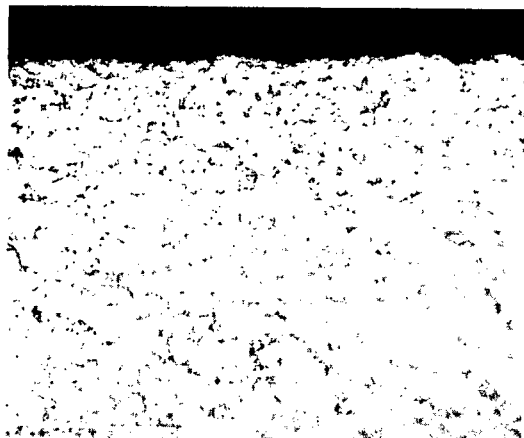


10 000 hours



15 000 hours

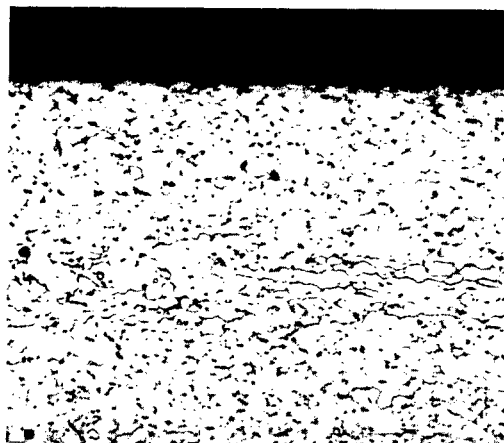
Figure 24.- Vibratory-treated self-stressed specimens after salt dip and various exposures at 600° F (590° K), as noted.
followed by room-temperature compression test. L-67-6689



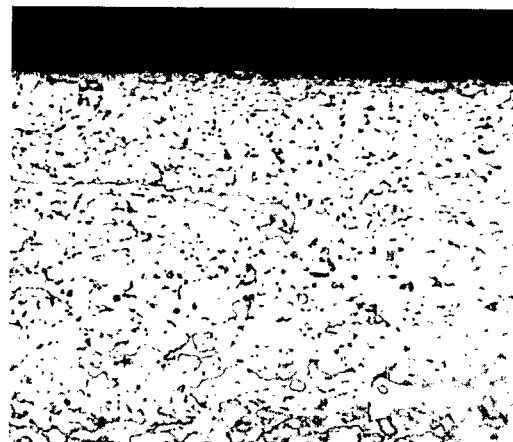
(a) Vibrated 4 hours; no exposure.



(b) Vibrated 26 hours; no exposure.



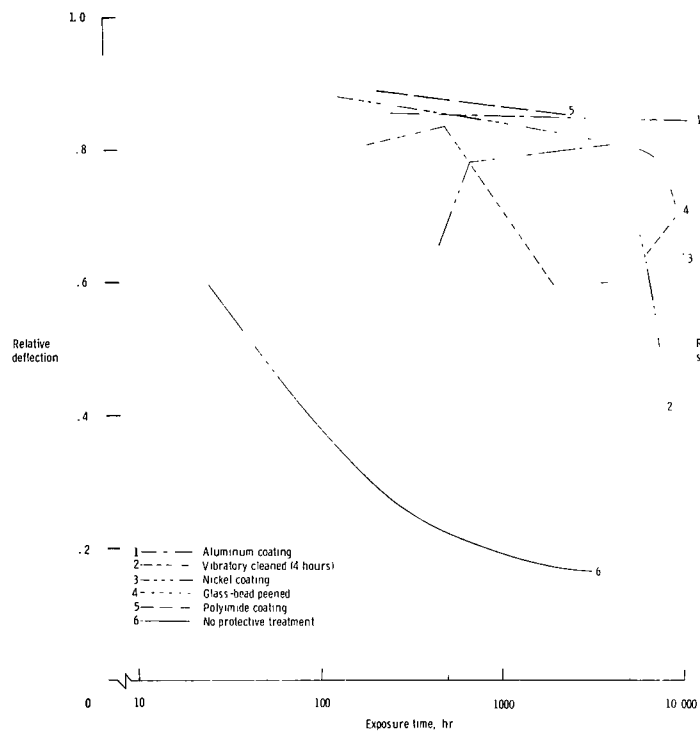
(c) Vibrated 4 hours; 6000 hours exposure.



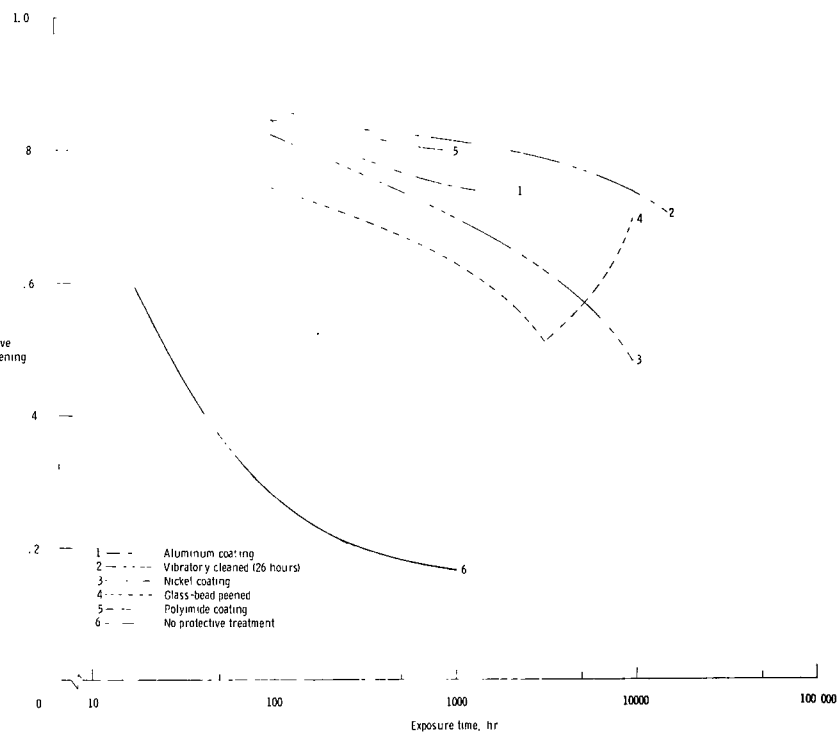
(d) Vibrated 4 hours; 8000 hours exposure.

L-67-6690

Figure 25.- Cross sections of vibratory-treated residually stressed specimens after various 600° F (590° K) exposures and room-temperature mechanical tests. X 500.



(a) Residual-stress specimens.



(b) Self-stressed specimens.

Figure 26.- Comparison of various protective treatments on salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet specimens after 600° F (590° K) exposure.

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